

UNCLASSIFIED

AD 268 438

*Reproduced
by the*

ARMED SERVICES TECHNICAL INFORMATION AGENCY
ARLINGTON HALL STATION
ARLINGTON 12, VIRGINIA



UNCLASSIFIED

NOTICE: When government or other drawings, specifications or other data are used for any purpose other than in connection with a definitely related government procurement operation, the U. S. Government thereby incurs no responsibility, nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use or sell any patented invention that may in any way be related thereto.

8
61-138
WADD TECHNICAL REPORT 61-138

THE EFFECT OF CONCURRENT STRAINING
AND A 1-PERCENT MAGNESIUM ADDITION
ON THE RECOVERY BEHAVIOR OF ALUMINUM

T. E. Tietz
C. L. Meyers
J. L. Lytton

Metallurgy and Ceramics Research
Lockheed Missiles and Space Division
Lockheed Aircraft Corporation

May 1961

Materials Central
Contract No. AF 33(616)-7156
Project No. 7351

62-5
XEROX



AERONAUTICAL SYSTEMS DIVISION
AIR FORCE SYSTEMS COMMAND
UNITED STATES AIR FORCE
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

NOTICES

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.



Qualified requesters may obtain copies of this report from the Armed Services Technical Information Agency, (ASTIA), Arlington Hall Station, Arlington 12, Virginia.



This report has been released to the Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C., for sale to the general public.



Copies of WADD Technical Reports and Technical Notes should not be returned to the Wright Air Development Division unless return is required by security considerations, contractual obligations, or notice on a specific document.

FOREWORD

This report was prepared by the Lockheed Missiles and Space Division, Lockheed Aircraft Corporation, under USAF Contract No. AF 33(616)-7156. This contract was initiated under Project No. 7351, "Metallic Materials," Task No. 73521, "Behavior of Metals." The project was administered under the direction of the Metals and Ceramics Laboratory, Materials Central, Directorate of Advanced Systems Technology, Wright Air Development Division, with Lt. Ben A. Wilcox acting as project engineer.

This report covers the period from 1 April 1960 to 1 February 1961.

The report was prepared as Technical Report No. LMSD-895071 by T. E. Tietz, C. L. Meyers, and J. L. Lytton, scientists in Metallurgy and Ceramics Research at LMSD. Acknowledgement is also made to R. J. Austin and R. W. Kelsey for their assistance during this program.

ABSTRACT

The effect of elastic strain, concurrent creep strain, and a 1-percent magnesium addition on the recovery behavior of a high-purity aluminum was investigated.

The degree of recovery of the prestrained test material was measured in terms of tensile flow stress at room temperature after recovery treatments between 80° and 200° C. Recovery behavior under no-load conditions was evaluated for the 99.995-percent aluminum and the Al-1%Mg alloy. The effect of concurrent elastic strain and creep straining during recovery of 99.995-percent aluminum was also studied.

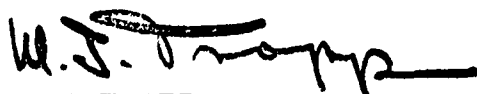
The activation energy for recovery of the 99.995-percent aluminum between 80° and 200° C was found to be $23,300 \pm 2,000$ calories per mole, and the activation energy for the Al-1%Mg alloy was 27,500 calories per mole.

Whereas concurrent elastic strain was concluded to have no effect on the rate of recovery of the 99.995-percent aluminum in terms of flow stress, concurrent creep straining had a very significant effect. However, the activation energy of the recovery process was not found to be significantly different as a result of concurrent creep straining.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:



W. J. TRAPP
Chief, Strength and Dynamics Branch
Metals and Ceramics Laboratory
Materials Central

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
1	INTRODUCTION	1
2	EXPERIMENTAL PROCEDURE	3
	2.1 Test Materials	3
	2.2 Apparatus	5
	2.3 Method of Evaluating Recovery	8
3	EXPERIMENTAL RESULTS	13
	3.1 Recovery Behavior of High Purity Aluminum	13
	3.2 Recovery Behavior of Al-1% Mg Alloy	17
	3.3 Recovery of High-Purity Aluminum Under Concurrent Elastic and Plastic Strain	28
4	DISCUSSION	37
	4.1 Previous Investigations	37
	4.2 No-Load Recovery of High-Purity Aluminum	38
	4.3 No-Load Recovery of Al-1% Mg Alloy	41
	4.4 Recovery Under Concurrent Elastic Strain	43
	4.5 Recovery of Cold-Worked High-Purity Aluminum During Concurrent Creep Straining	44
5	CONCLUSIONS	49
6	REFERENCES	51

LIST OF ILLUSTRATIONS

<u>Figure</u>		<u>Page</u>
1	Tensile Test Specimen Design	4
2	Tensile Specimen and Extensometer Assembly in Test Position	6
3	Constant Temperature Baths Used for Recovery Under No-Load Conditions	7
4	Experimental Setup for Studying Recovery Behavior Under Conditions of Concurrent Stress	9
5	Schematic Diagram Showing the Effect of Recovery on the Stress-Strain Curve and the Method Used To Evaluate the Degree of Recovery	10
6	Effect of Recovery Treatment at 80° C on the True Stress-True Strain Curve of 99.995-Percent Aluminum	14
7	Effect of Recovery Treatment at 201° C on the True Stress-True Strain Curve of 99.995-Percent Aluminum	15
8	Fractional Recovery as a Function of Recovery Time for 99.995-Percent Aluminum for Four Recovery Temperatures	16
9	Equivalence of Recovered State for Four Pairs of 99.995-Percent Aluminum Specimens Which Exhibited Constant Values of Fractional Recovery	18
10	Effect of Recovery Treatment at 80° C on the True Stress-True Strain Curve of Al-1% Mg Alloy	19
11	Effect of Recovery Treatment at 201° C on the True Stress-True Strain Curve of Al-1% Mg Alloy	20
12	Fractional Recovery as a Function of Recovery Time for Al-1% Mg Alloy for Four Recovery Temperatures	22
13	Comparison of Fractional Recovery as a Function of Recovery Time for Both 99.995-Percent Aluminum and Al-1%Mg Alloy for Four Recovery Temperatures	23
14	Stress-Strain Curves Showing Equivalence of Recovered States at Constant Fractional Recovery for Al-1%Mg Alloy	25
15	Effect of Prestrain on the Stress-Strain Curve of 99.995-Percent Aluminum After Recovery for 41 Hr at 160° C	26

<u>Figure</u>		<u>Page</u>
16	Effect of Prestrain on the Stress-Strain Curve of Al-1%Mg Alloy After Recovery for 6.0 Hr at 160° C	27
17	Effect of Concurrent Elastic Strain on Recovery of 99.995-Percent Aluminum at 80° and 198° C	29
18	Typical Creep Curves for 99.995-Percent Aluminum After Pre-straining to 0.10 at Room Temperature	31
19	Effect of Concurrent Creep Straining on Recovery of 99.995-Percent Aluminum at 160° and 198° C Under the Same Stress	32
20	Equivalence of Recovered States at Constant Fractional Recovery During Concurrent Creep Straining	33
21	Effect of Concurrent Creep Straining on Recovery of 99.995-Percent Aluminum at 80° and 198° C Under Various Stresses	34
22	Time Temperature Correlation of Fractional Recovery of 99.995-Percent Aluminum Using $\theta = t e^{-Q/RT}$	40
23	Time-Temperature Correlations of Fractional Recovery for 99.995-Percent Aluminum for Both Conditions of No-Load and Concurrent Straining	46
24	Fractional Recovery Due to Creep as a Function of Creep Strain for 99.995-Percent Aluminum	48

Section 1
INTRODUCTION

During cold work a number of properties of metals change, which upon heating to elevated temperatures recover toward their original values for the unstrained state. For the purposes of this investigation any process, short of recrystallization, which contributes to this return of properties will be called a recovery process. Thus the degree of recovery of a cold-worked metal may be studied by measuring the changes which take place in any of a number of properties. Some of the properties which have been used by past investigators include X-ray diffraction phenomena, electrical resistivity, hardness, creep strength, elastic limit, and flow stress. An excellent review of the recovery of mechanical properties has been published by Perryman.⁽¹⁾

Despite the apparent preponderance of studies made to date on recovery behavior, there still remain many important questions which must be answered to allow intelligent selection of alloy additions and thermal-mechanical treatments for elevated temperature applications. Two of these areas which preliminary observations indicate should have an important influence on the rate of recovery and which need clarification are the effect of solid-solution alloy additions and the effect of concurrent straining during the recovery process.

A number of investigators⁽¹⁾ have demonstrated that impurities affect the rate of recovery and the substructures formed during recovery. Limited studies have indicated that in some cases solid-solution additions greatly decrease the rate of recovery. However, in at least the case of magnesium additions to aluminum, an increase

Manuscript released by the authors 24 February 1961 for publication as a WADD Technical Report.

in the rate of recovery has been reported. ⁽²⁾ These observations indicate that a very important aspect of alloy strengthening at elevated temperatures, in addition to the increased flow stress necessary to move dislocations through alloys, is the effect alloy additions have on the recovery behavior. To date, little work has been done to elucidate the influence that specific interstitial and substitutional additions have on the recovery processes.

Since metals in structural applications at elevated temperatures are under stress and often experience creep straining, it is apparent that the effects of concurrent elastic and plastic straining on the rate of recovery of cold-worked metals is of practical importance. Although some studies have been made in this area, they have been primarily in terms of resistivity changes, ⁽³⁾ and structural changes. ⁽⁴⁾ In terms of mechanical properties, Sherby et al. ⁽⁵⁾ observed that the rate of recovery of aluminum prestrained at liquid nitrogen temperatures as measured by tensile properties was greater during creep under an applied load than for an equal treatment under no load. Rinnoivore and Brown ⁽⁶⁾ found that plastic yielding during recovery of zinc single crystals inhibited the recovery process as measured by flow stress. In neither case, however, were the kinetics of the phenomena investigated.

The purpose of the current study was to increase our understanding of the effect of concurrent straining and the effect of alloying by evaluating (1) the effect of elastic strains and concurrent creep strains on the recovery of high-purity aluminum and (2) the effect of a 1-percent magnesium addition on the recovery of high-purity aluminum. Because of the particular interest in the effect of these factors on the recovery behavior as related to the mechanical properties, the degree of recovery was evaluated in terms of tensile properties.

Section 2
EXPERIMENTAL PROCEDURE

2.1 TEST MATERIALS

The materials used in this study were a high-purity aluminum and a solid-solution aluminum-magnesium alloy, both obtained from the Alcoa Research Laboratory, New Kensington, Pennsylvania. Analyses for these materials are presented in Table I. Both materials were received in the form of 0.125-in. thick sheets, as cold-rolled. Tensile specimens were cut in the rolling direction from the as-received sheets, using a Tensilkut high-speed pin router and template to produce the specimen geometry shown in Fig. 1. The recrystallization treatment and resulting grain size for each of the test materials is given in Table II.

Table I
CHEMICAL ANALYSIS OF TEST MATERIAL

<u>Material</u>	<u>Alcoa Lot No.</u>	Al	Cu	Fe	Si	Mg	Others
99.995% Al	197696	99.995	0.001	0.002	0.001	0.0004	0.000
Al-Mg Alloy	197072	99.00	0.001	0.002	0.003	0.99	0.000

Table II
RECRYSTALLIZATION TREATMENT

<u>Material</u>	<u>Annealing Treatment in Salt Bath</u>	<u>Recrystallized Grain Size (mm)</u>
99.995% Al	20 min at 450°C	0.4
Al - 1% Mg	20 min at 500°C	0.3

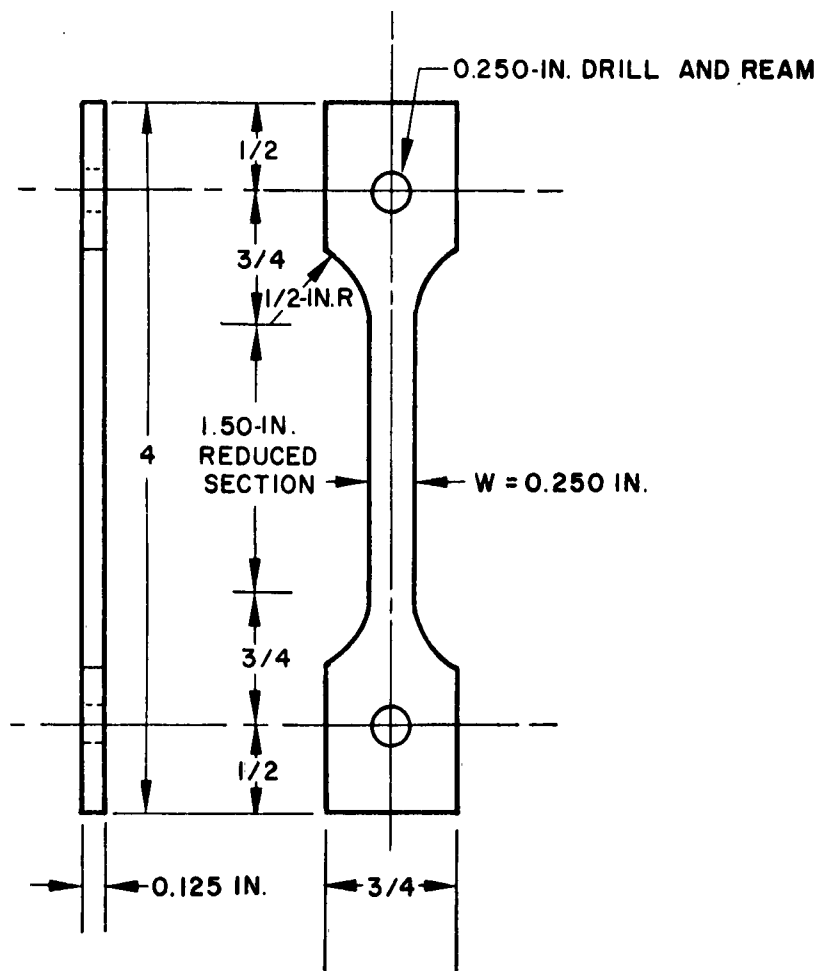


Fig. 1 Tensile Test Specimen Design

2.2 APPARATUS

The equipment used in this investigation consisted basically of three parts: (1) a means of performing and recording tensile stress-strain behavior at room temperature, (2) controlled temperature baths for recovery treatments of the prestrained specimens, and (3) equipment for application of various stress levels during recovery, and for measurement of any creep strains which occurred.

The room-temperature straining of specimens, before and after recovery treatment, was accomplished with an Instron Testing machine which autographically recorded the load-deflection curve, using a 1000-lb load cell. All tensile tests, prior to recovery and after recovery, were conducted at room temperature at a strain rate of about 0.03 per minute. An assembly of the tensile specimen and extensometer, mounted in the Instron machine, is shown in Fig. 2. The extensometer consists of two linear-variable-differential transformers (LVDT's) mounted on the pull-bar with one transformer core connected to the upper gage block and the other core to the lower gage block. The net output of the two transformers was proportional to the extension between the two gage blocks. This output was amplified within the Instron and recorded autographically. The resulting least counts in the autographically recorded load-deformation curve were equivalent to a strain of 0.002, and a stress of 60 psi in the case of the high-purity aluminum and of 150 psi in the case of the Al-Mg alloy.

Figure 3 shows the constant-temperature baths used for the recovery treatments under no-load conditions. Four recovery baths were used at nominal temperatures of 80°, 120°, 160°, and 200°C. Cottonseed oil was used for the three lower-temperature baths, and silicone oil for the 200°C bath. The bath temperatures were held constant by means of a mercury thermoregulator. For the majority of the recovery tests the bath temperatures were held constant to within $\pm 1^\circ\text{C}$. However, in a few cases variations as great as $\pm 2^\circ\text{C}$ occurred.

For the purpose of investigating the effect of concurrent elastic strain and plastic strain upon recovery, two additional recovery baths were mounted on constant-load

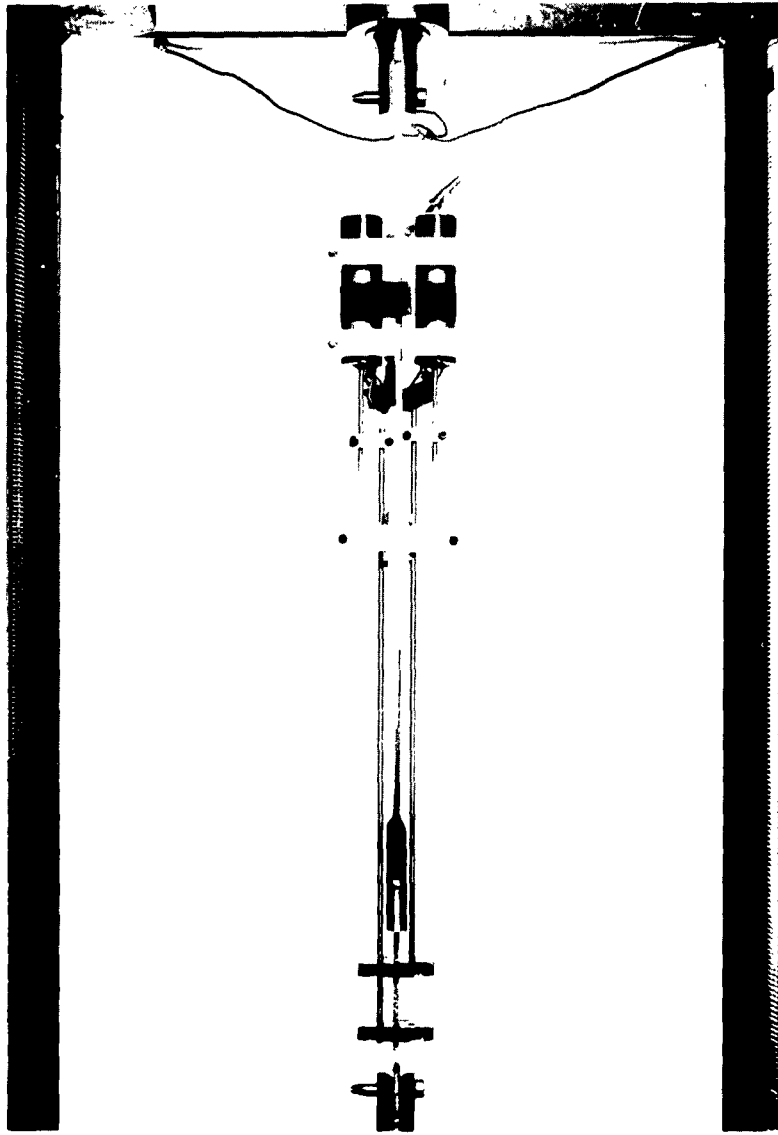


Fig. 2 Tensile Specimen and Extensometer Assembly in Test Position

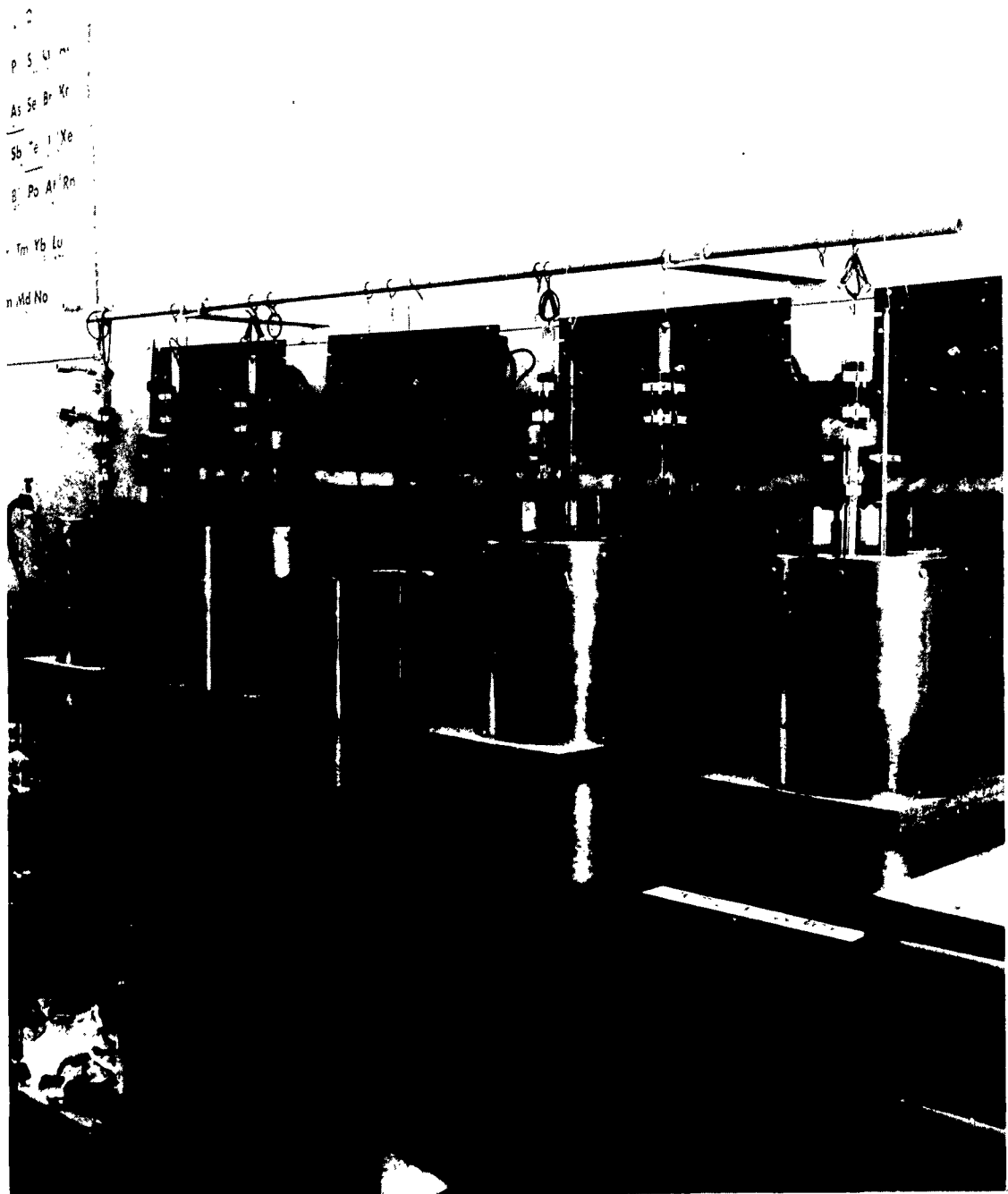


Fig. 3 Constant Temperature Baths Used for Recovery Under No-Load Conditions

creep frames, as shown in Fig. 4. These baths were mounted on drill press columns so they could be raised to immerse the test specimen in the oil. Creep strain achieved during recovery was determined by exciting the LVDT's with a constant voltage input and measuring the diode-rectified output on a D-C millivoltmeter. This system was extremely sensitive, but fluctuation of the background signals picked up by the shielded circuit caused the effective least count to be equivalent to a strain of about 0.002, or about equal to that used on the Instron recorder. Specimens were loaded through 20-to-1 lever arms prior to application of the oil bath, and were unloaded after oil quenching to near room temperature.

At the outset of this program, a specimen was mounted in an extensometer and a thermocouple was spot-welded to the center of the gage section. Heating curves were obtained for each bath temperature so that the time-temperature path for the specimen to reach bath temperature was determined. After the results of longer time tests had indicated the magnitude of the activation energy for recovery in the 80° to 200°C temperature range, it was possible to calculate accurately the correction in recovery time necessary to subtract to obtain the total effective time at bath temperature. This time correction ranged from 1.06 min to 1.8 min, and was significant for only the shorter recovery treatments of 1 hr or less. The quenching operation was found to be rapid enough so that no correction was necessary for this operation.

2.3 METHOD OF EVALUATING RECOVERY

The degree of recovery after prestraining at room temperature was evaluated in terms of tensile properties by measuring the decrease in initial flow stress after various recovery treatments. Figure 5 shows schematically the effect of a recovery treatment on the true stress-true strain, σ - ϵ , curve. The degree of recovery was evaluated in terms of fractional recovery f_r , where f_r is defined as the decrease in the flow stress due to the recovery treatment divided by the decrease experienced if the material recovered completely to the unstrained state. Thus, as illustrated in Fig. 5,

$$f_r = \frac{(\sigma_1 - \sigma_2)}{(\sigma_1 - \sigma_Y)}$$

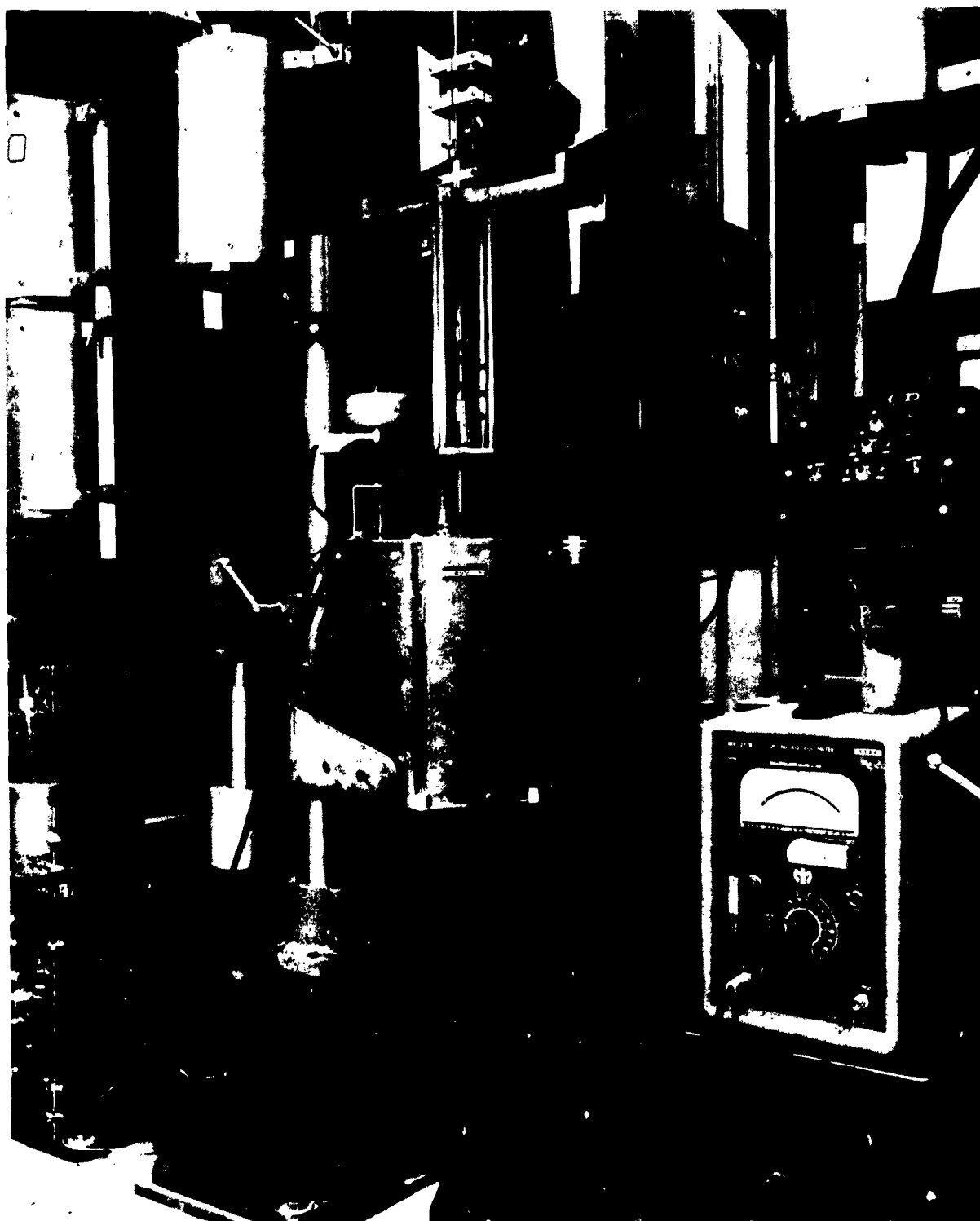


Fig. 4 Experimental Setup for Studying Recovery Behavior Under Conditions of Concurrent Stress

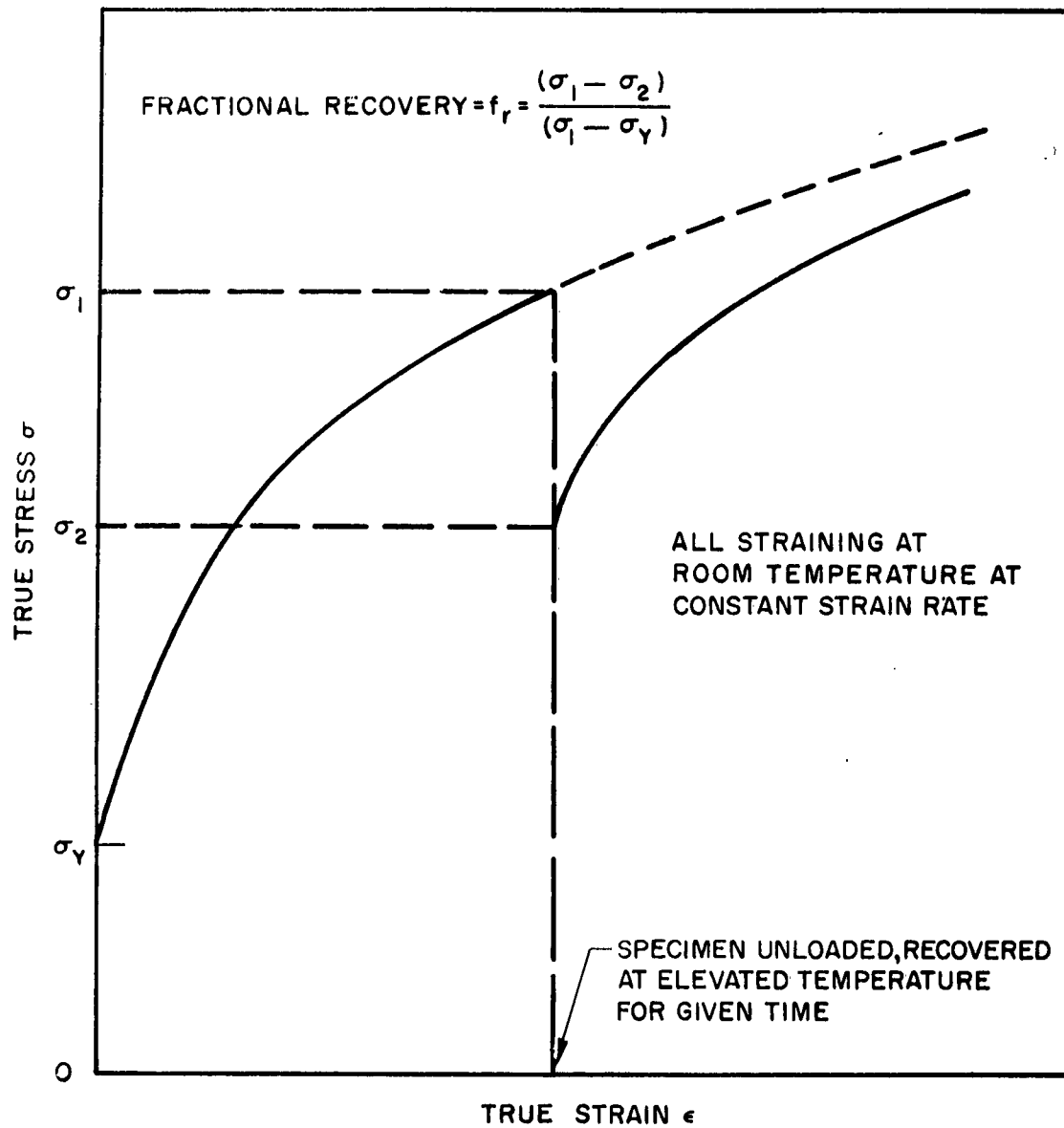


Fig. 5 Schematic Diagram Showing the Effect of Recovery on the Stress-Strain Curve and the Method Used To Evaluate the Degree of Recovery

In the case of the high-purity aluminum the initial yield stress, σ_Y , upon straining the recrystallized material was very low, in some cases approaching zero. The values varied, apparently due to slight differences in handling the very soft material during mounting the extensometer. These slight differences in initial yield strength appeared to have little if any effect upon the subsequent flow-stress curve. In calculating f_r for the recovery tests on the high-purity aluminum, σ_Y was thus taken as zero. In the case of the Al-Mg alloy the initial yield strength was relatively high and fairly constant at a value of about 3000 psi. In calculating f_r for the alloy, the actual value of σ_Y was used.

Several specimens were prestrained, unloaded for a short time at room temperature and restrained. A slight drop was observed in the initial yield strength about equal to the width of the recorded ink line. This drop, attributed to a small amount of recovery at room temperature, was equal to an f_r value of about 0.003.

Laue back-reflection studies which were conducted on the high-purity aluminum after 15-percent prestrain showed that no recrystallization was obtained after 1 hr at 450°C, indicating that only recovery occurred at the highest temperature of 200°C used in this investigation. Although no recrystallization occurred after 1 hr at 450°C for a specimen prestrained 15 percent, the as-received material which was in a severely cold-worked condition as indicated earlier was completely recrystallized at the same temperature in 20 minutes.

Section 3

EXPERIMENTAL RESULTS

All tensile data, before and after recovery treatment, were obtained at room temperature in the form of autographic recordings of load-deformation curves. The true stress-true strain curves presented in this section are based upon selected values taken from the continuous curves which were recorded. The technique of evaluating the degree of recovery in terms of the decrease in initial flow stress after the recovery treatment proved very satisfactory and resulted, in most cases, in a well-defined initial flow stress. In the most poorly defined cases, which were for the longer recovery times, the uncertainty in the initial flow stress was still less than ± 5 percent.

3.1 RECOVERY BEHAVIOR OF HIGH-PURITY ALUMINUM

The effect of recovery treatment at 80°C on the true stress-true strain curve of the 99.995-percent aluminum is presented in Fig. 6. The upper curve represents the $\sigma - \epsilon$ curve for a continuous test without a recovery treatment. The other three curves represent specimens which were prestrained at room temperature to 10-percent true strain, unloaded and recovered at 80°C for times of 0.07, 4.0, and 100 hr, and finally poststrained at room temperature. A drop in the entire stress-strain curve is observed; this drop increases with increasing recovery time at 80°C .

Figure 7 presents similar stress-strain curves for the high-purity aluminum after various recovery times at 201°C .

The recovery data for high-purity aluminum are summarized in Fig. 8 for the four recovery temperatures in terms of the fractional recovery f_r versus the log of the recovery time. The data result is straight, almost parallel lines with the exception of the shorter recovery times at the lowest recovery temperature, 80°C .

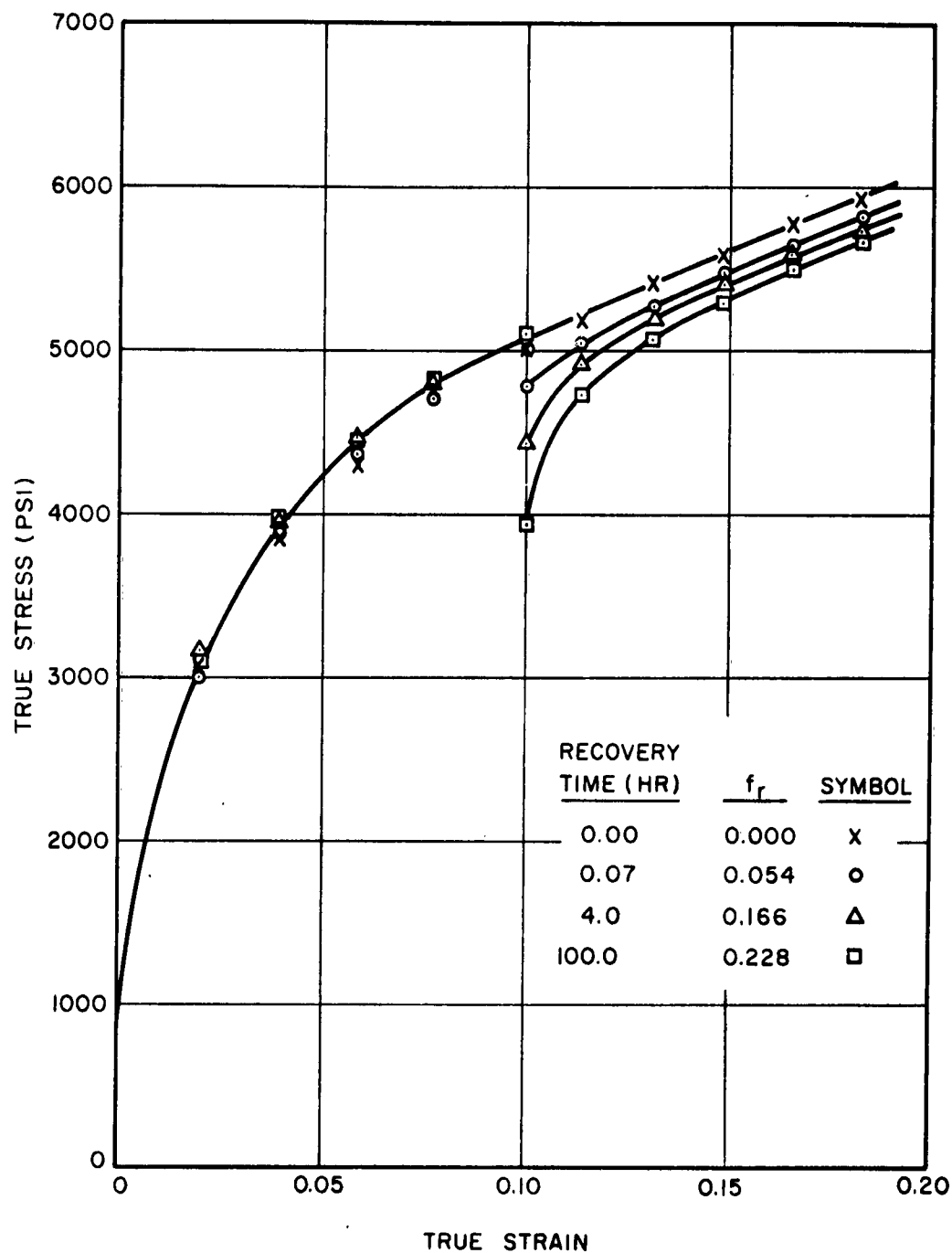


Fig. 6 Effect of Recovery Treatment at 80°C on the True Stress-True Strain Curve of 99.995-Percent Aluminum

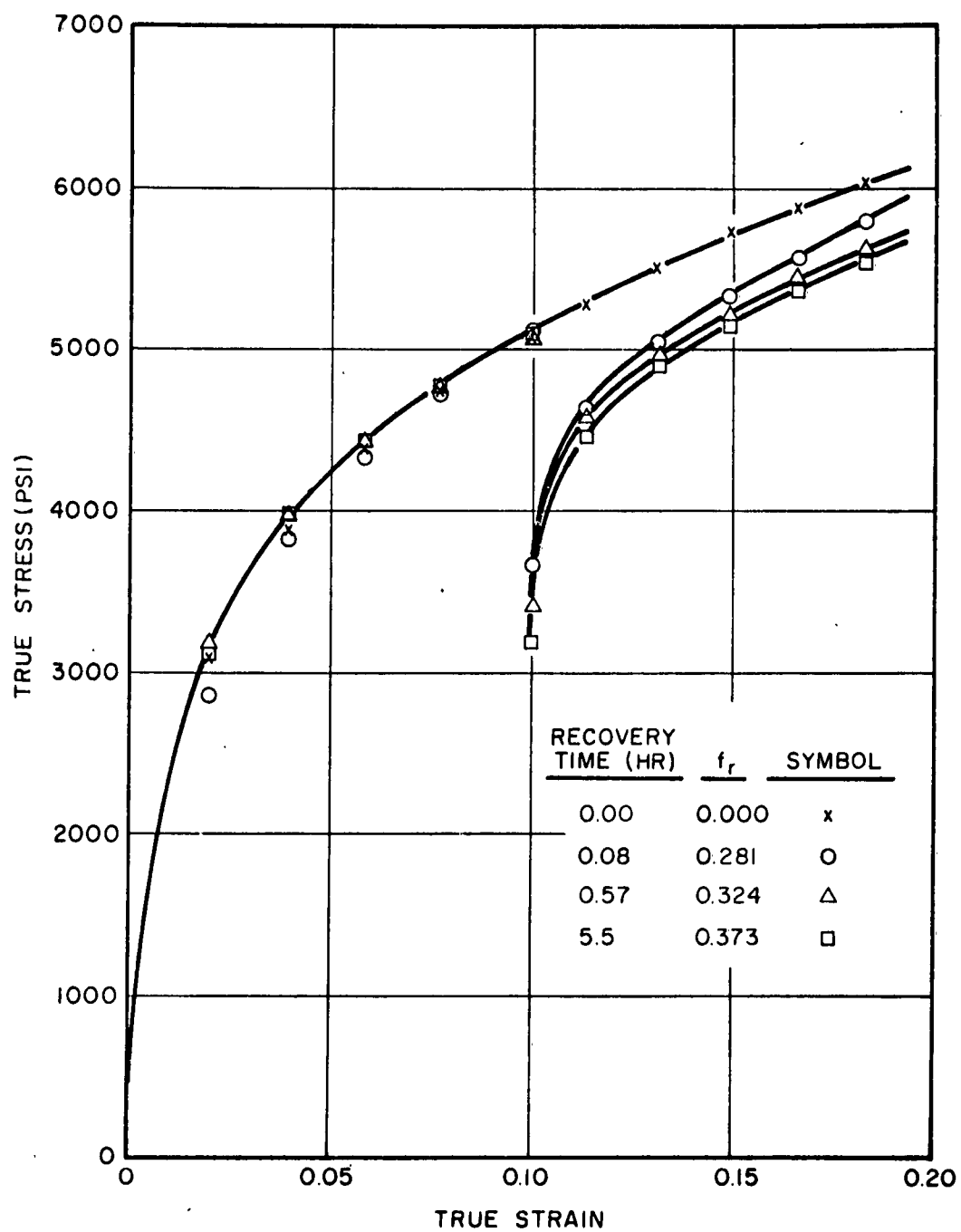


Fig. 7 Effect of Recovery Treatment at 201° C on the True Stress-True Strain Curve of 99.995-Percent Aluminum

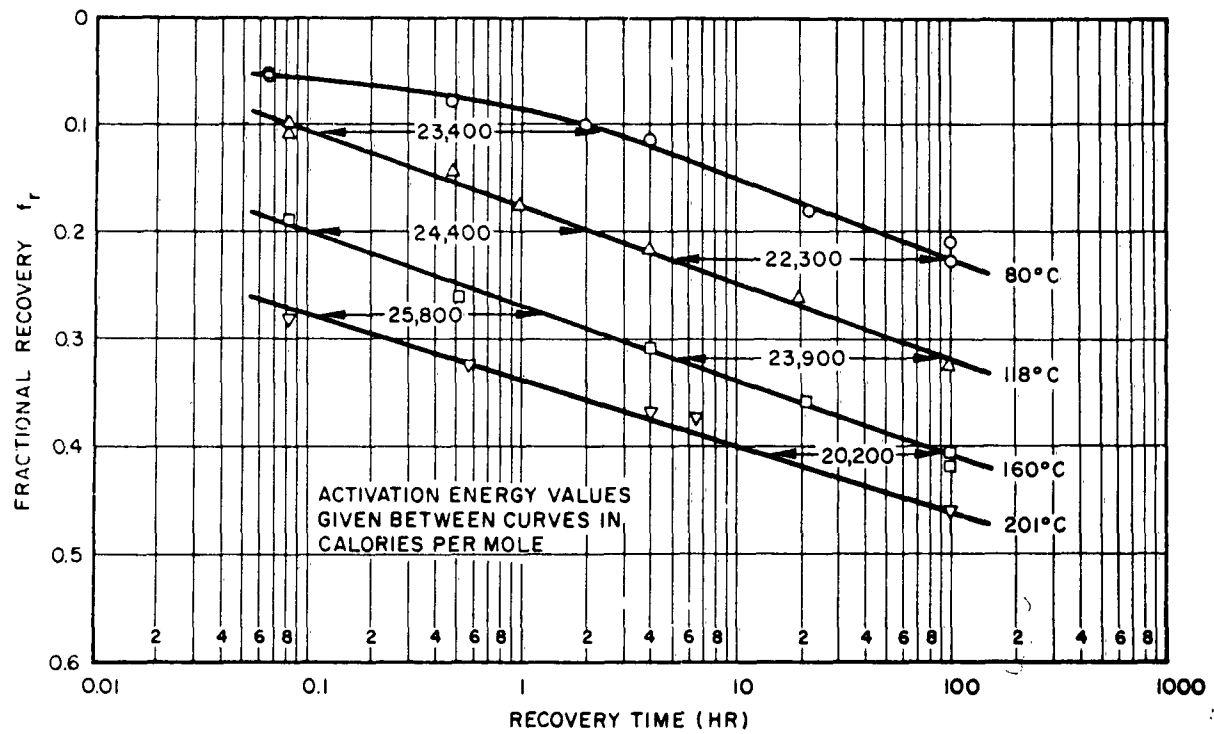


Fig. 8 Fractional Recovery as a Function of Recovery Time for 99.995-Percent Aluminum for Four Recovery Temperatures

To calculate the activation energy for the recovery process, information regarding the existence of equivalent states of the material between two recovery temperatures must be known or assumed. It has been the practice of past investigators to assume that equivalent states were obtained during recovery at different temperatures when the amounts of recovery of the particular property being measured were equal. The present investigators set out to demonstrate whether this assumption is valid. The initial intent was to check the equivalency of the recovered states for a pair of specimens which exhibited equal values of f_r after recovery at different temperatures, by comparing the entire stress-strain curve after recovery. Superposition of the two stress-strain curves would indicate that not only were the values of the initial flow stress equal but also the rates of strain hardening were identical for both cases, or that equivalent structural states existed. However, to achieve equal recovered states, even for the case of two specimens given identical recovery treatments, it is necessary that the initial strain-hardened states prior to recovery be equal. Because of experimental scatter of the stress-strain curves during prestraining, it was usually not possible to obtain equal strain-hardened states.

For this reason, comparison was made of four pairs of tests, each of which had equal values of f_r , on the basis of percent of true stress at 0.10 true strain. These comparisons are presented in Fig. 9. The superposition of the curves in most cases is quite good and appears to substantiate the assumption of equivalent structural states at equal values of f_r .

3.2 RECOVERY BEHAVIOR OF Al-1% Mg ALLOY

The effect of recovery treatment at 80°C on the true stress-true strain curve of the Al-1% Mg alloy is illustrated in Fig. 10. It will be noted that there is very little additional drop in the stress-strain curve because of extending the recovery time at 80°C from 100 to 500 hr. Figure 11 presents similar recovery data for the alloy after various recovery times, at 201°C. Of particular interest here is the rapid initial rate of recovery. A 5-min recovery treatment resulted in a drop in the

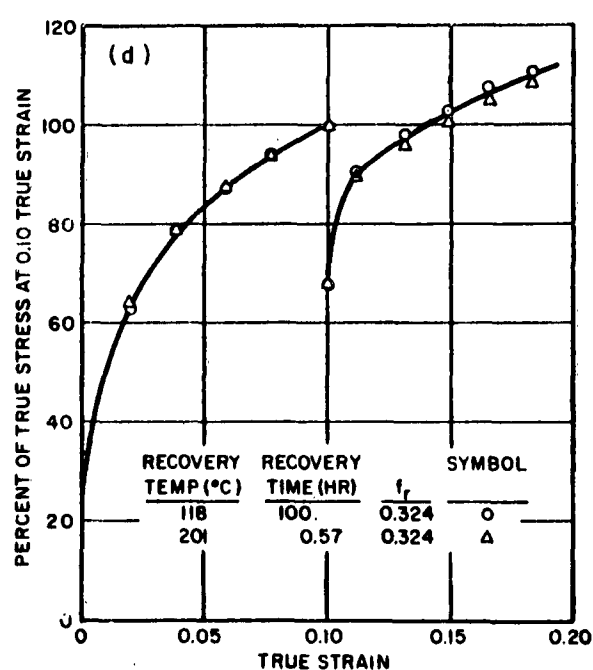
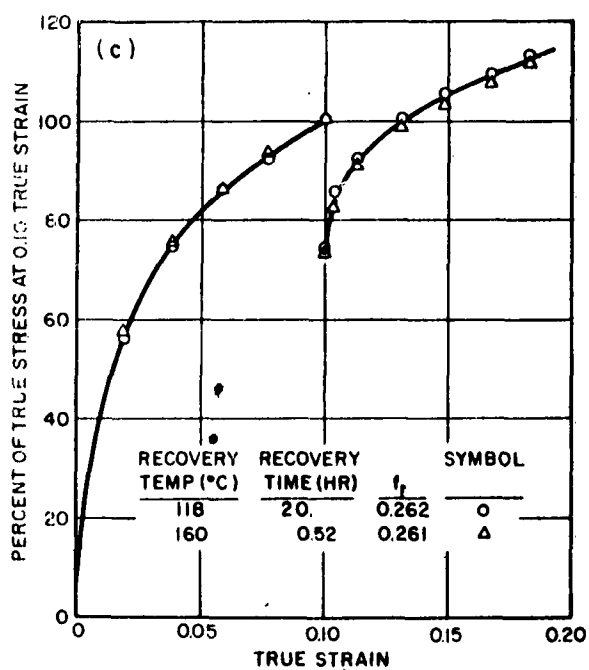
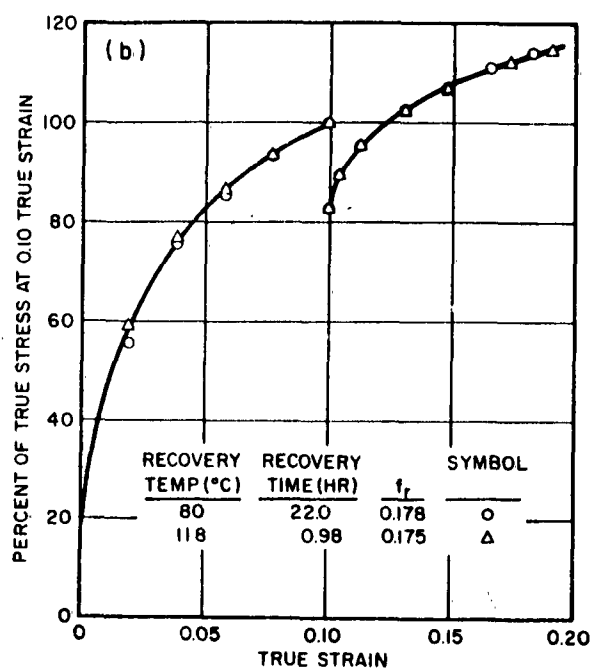
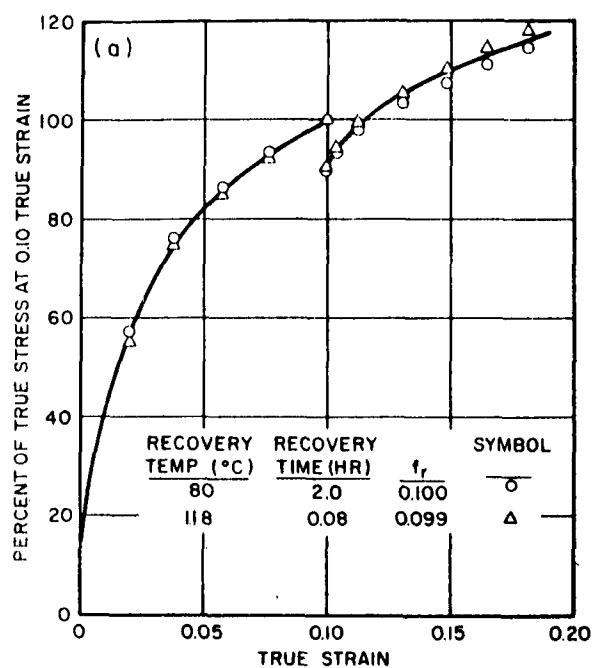


Fig. 9 Equivalence of Recovered State for Four Pairs of 99.995-Percent Aluminum Specimens Which Exhibited Constant Values of Fractional Recovery

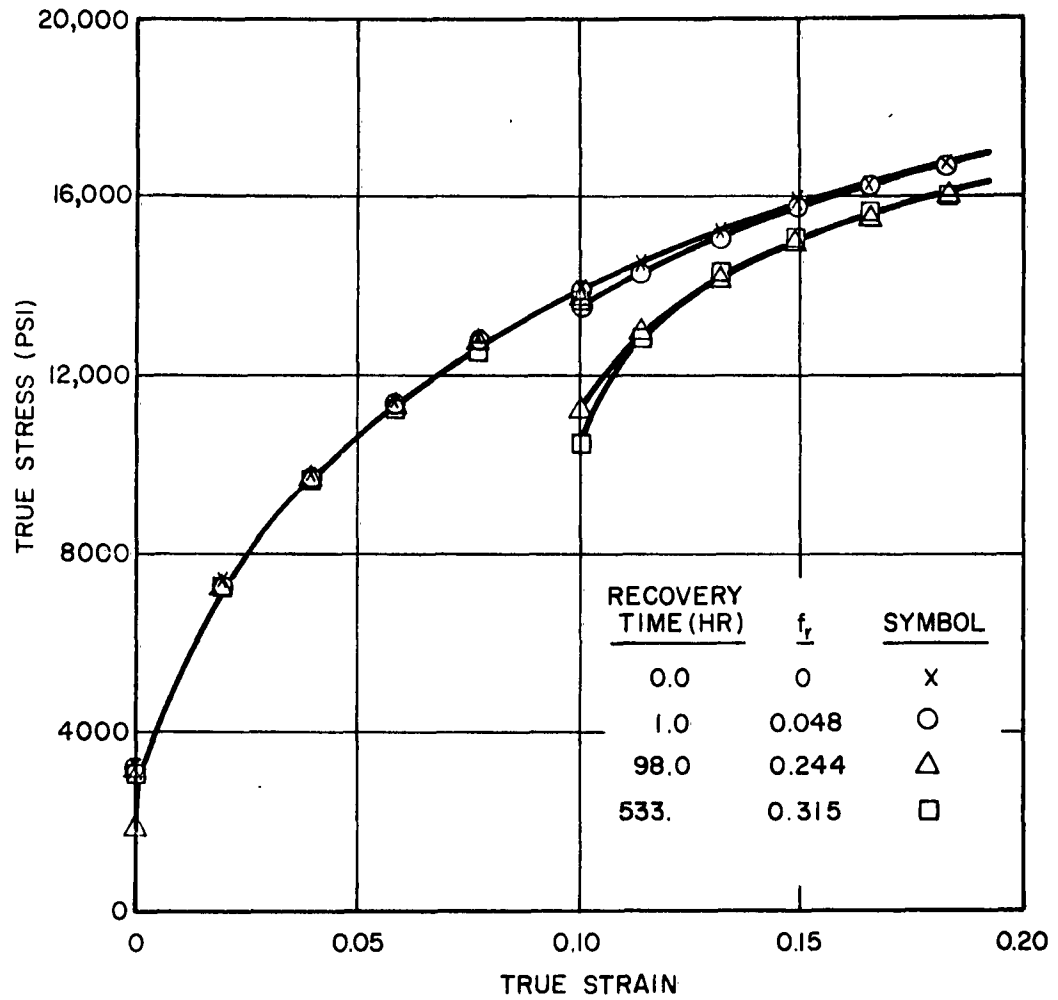


Fig. 10 Effect of Recovery Treatment at 80°C on the True Stress-True Strain Curve of Al-1%Mg Alloy

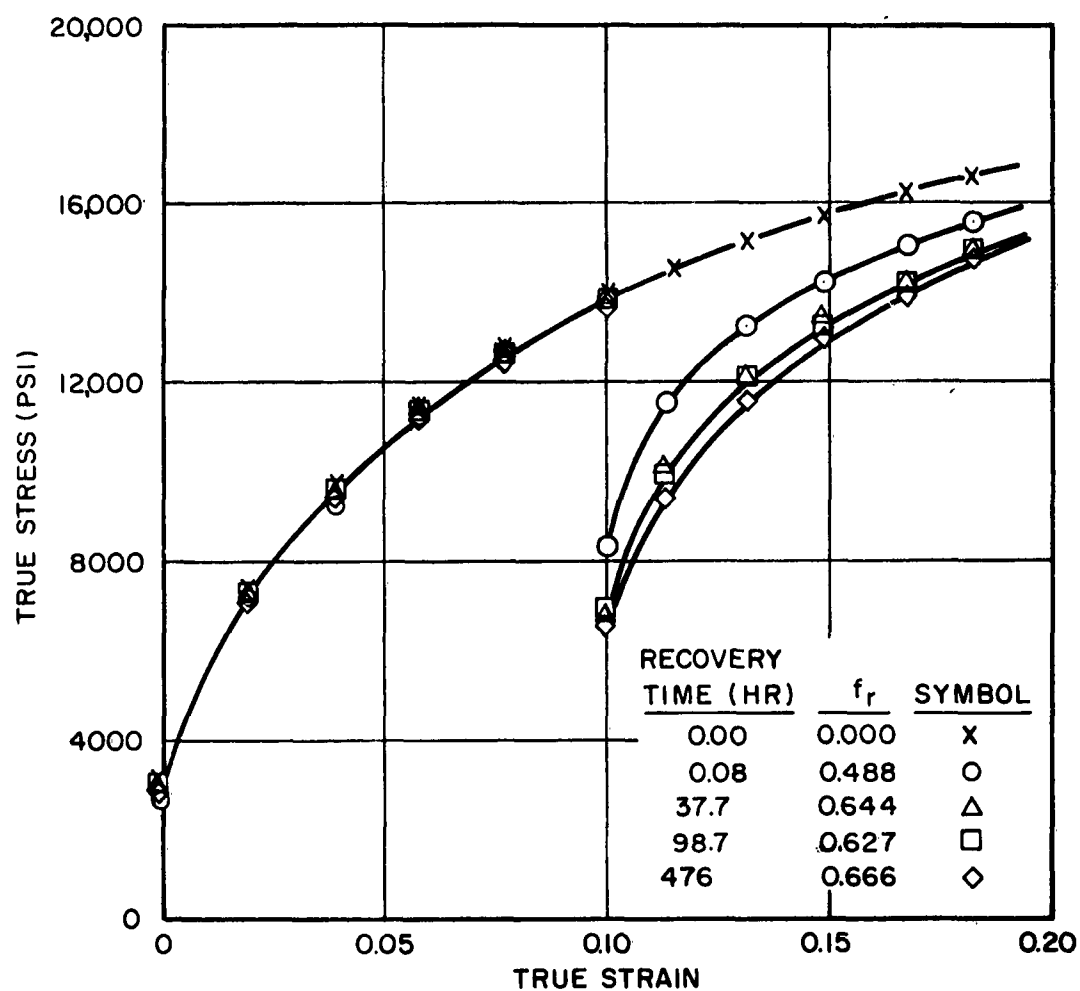


Fig. 11 Effect of Recovery Treatment at 201°C on the True Stress-True Strain Curve of Al-1% Mg Alloy

initial flow stress in terms of fractional recovery of 0.5, whereas an additional recovery time of almost 500 hr results in only an additional fractional recovery of 0.16.

Figure 12 summarizes the recovery data for the Al-Mg alloy for the four recovery temperatures in terms of fractional recovery f_r as a function of the log of the recovery time. To allow a direct comparison between the recovery data for the high-purity aluminum and the Al-Mg alloy, the curves of Figs. 8 and 12 have been combined in Fig. 13. A number of observations are of interest:

- At 80°C the alloy initially exhibited less recovery in terms of f_r than the high-purity aluminum, but after about a 30-hr recovery time the alloy experienced a larger fractional recovery than the aluminum.
- At 118°C the alloy recovered less initially than the high-purity aluminum, but the slope of the f_r versus $\log t$ curve for the alloy is much greater and after only 0.2 hr the f_r value for the alloy was higher than that for the aluminum.
- At 160°C at 0.1 hr the alloy already recovered more than the high-purity aluminum at the same temperature.
- The slopes of the linear portion of the f_r versus $\log t$ curves at 80°, 118°, and 160°C are about parallel and are greater for the alloy than for the high-purity aluminum.
- At 201°C the alloy experienced considerably more recovery than the aluminum, about twice the fractional recovery at 0.1 hr; however, the slope of the initial part of the f_r versus $\log t$ curve for the alloy was less at the 201°C recovery temperature than at the lower three temperatures. In fact, at 201°C the slope of the curve for the alloy is about identical to the slope of the f_r versus $\log t$ curves for the high-purity aluminum.
- An additional item of interest is the definite break in the f_r versus $\log t$ curves for the alloy for the higher three recovery temperatures, where the fractional recovery of flow stress appears to stop.

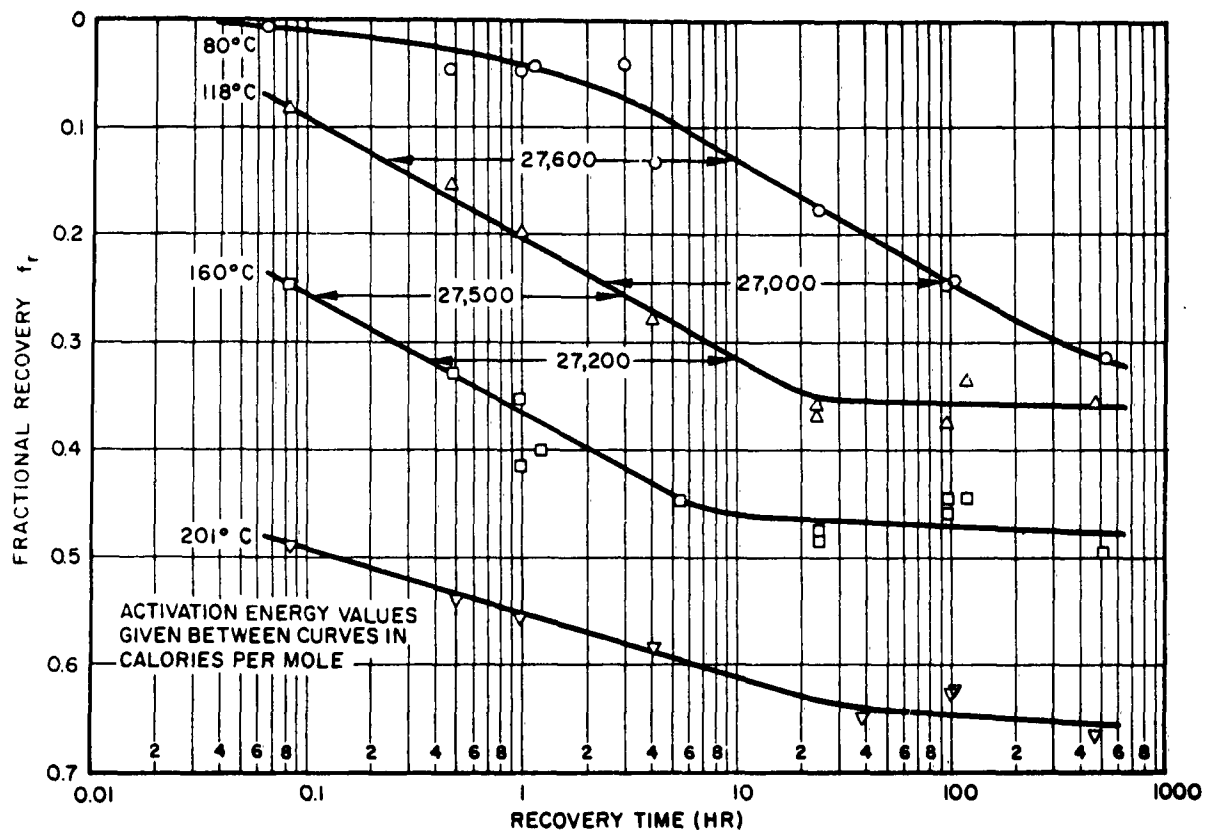


Fig. 12 Fractional Recovery as a Function of Recovery Time for Al-1% Mg Alloy for Four Recovery Temperatures

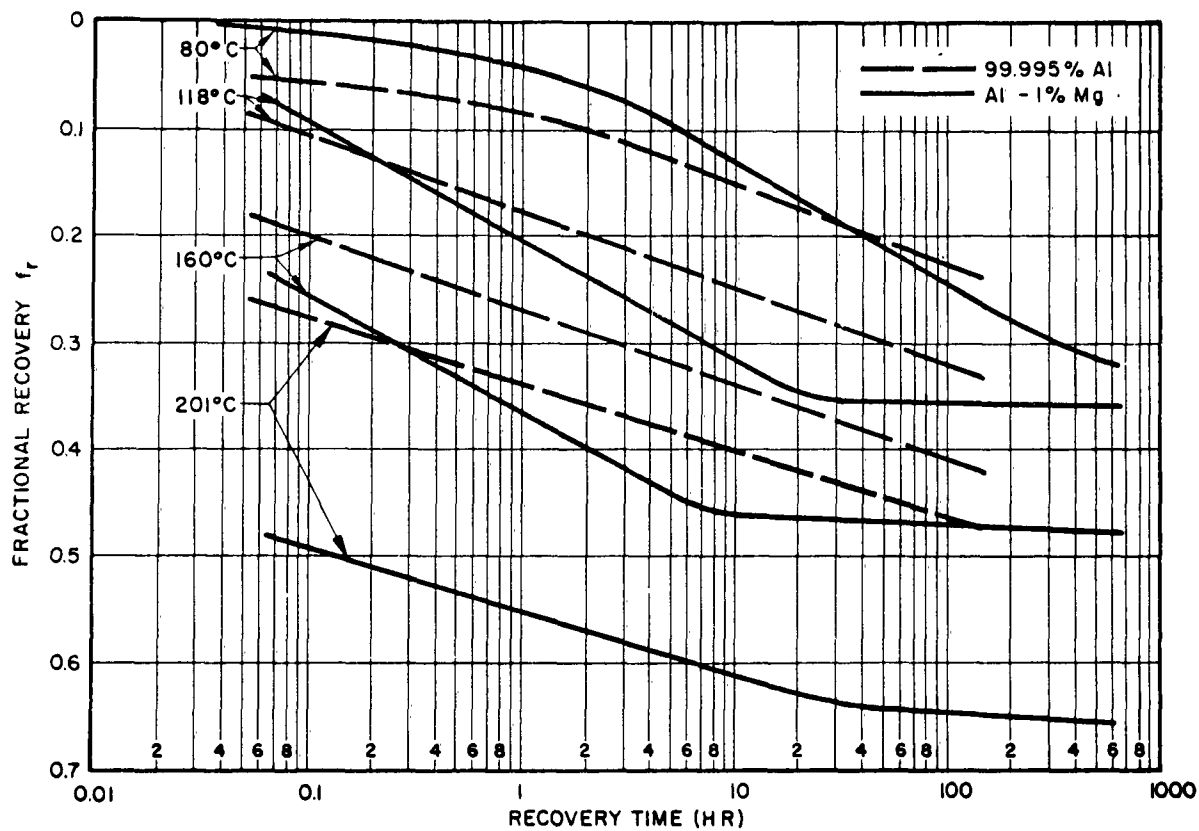


Fig. 13 Comparison of Fractional Recovery as a Function of Recovery Time for Both 99.995-Percent Aluminum and Al-1%Mg Alloy for Four Recovery Temperatures

The data for the alloy were examined for tests having almost identical values of f_r in an effort to check whether equivalent structural states were obtained independent of the recovery temperature. Two tests had an f_r value of 0.25, one after 5 min at 160°C and one after 98 hr at 80°C. Since these two specimens gave very nearly identical $\sigma - \epsilon$ curves prior to recovery, it was possible to compare the tests on the basis of the actual $\sigma - \epsilon$ curves rather than on the basis of the percentage of flow stress. The superposition of the stress-strain curves for these two tests before and after recovery is very good, as shown in Fig. 14. Since the initial flow stress after recovery and the rate of work hardening are identical, both properties being structure-sensitive, it is concluded that the two specimens which were recovered at different temperatures to a constant value of f_r had equivalent structural states.

On this basis, activation energy values calculated for the recovery process for the alloy were about 27,500 calories per mole over the temperature range of 80° to 160°C, as indicated in Fig. 12.

All the recovery tests presented so far were conducted on specimens prestrained to a true strain of 0.10. This results, of course, in different work-hardened states for the high-purity aluminum and the Al-Mg alloy. Reference to Figs. 7 and 11 indicates that a true stress of about 5,000 psi was required to continue to deform the high-purity aluminum at 0.10 strain, whereas about 14,000 psi was required in the case of the Al-Mg alloy. This poses a question as to whether the degree of prior work-hardening in either material would change the relative recovery behavior for the high-purity aluminum and the Al-Mg alloy. In an attempt to clarify this factor, a series of recovery tests were conducted at 160°C on both test materials to determine the effect of different amounts of prestrain on the recovery behavior. The results of these tests are presented in Fig. 15 for the high-purity aluminum and in Fig. 16 for the Al-Mg alloy. The value of f_r for the high-purity aluminum after a 41-hr recovery at 160°C appears to be independent of prestrain between a strain of 0.04 and 0.14 and has a value of about 0.39. The fact that the values of f_r are equal does not imply, of course, that the structural states are equal after recovery

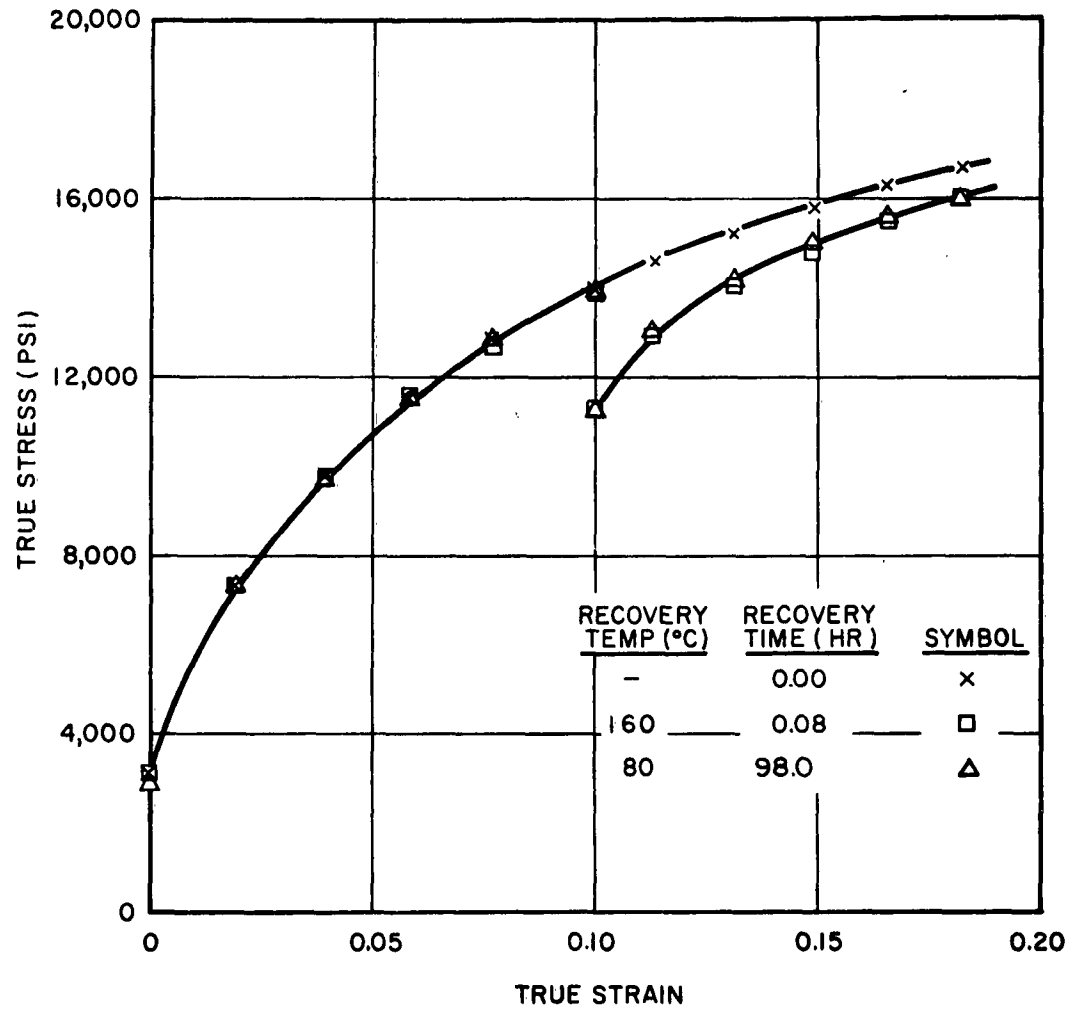


Fig. 14 Stress-Strain Curves Showing Equivalence of Recovered States at Constant Fractional Recovery for Al-1%Mg Alloy

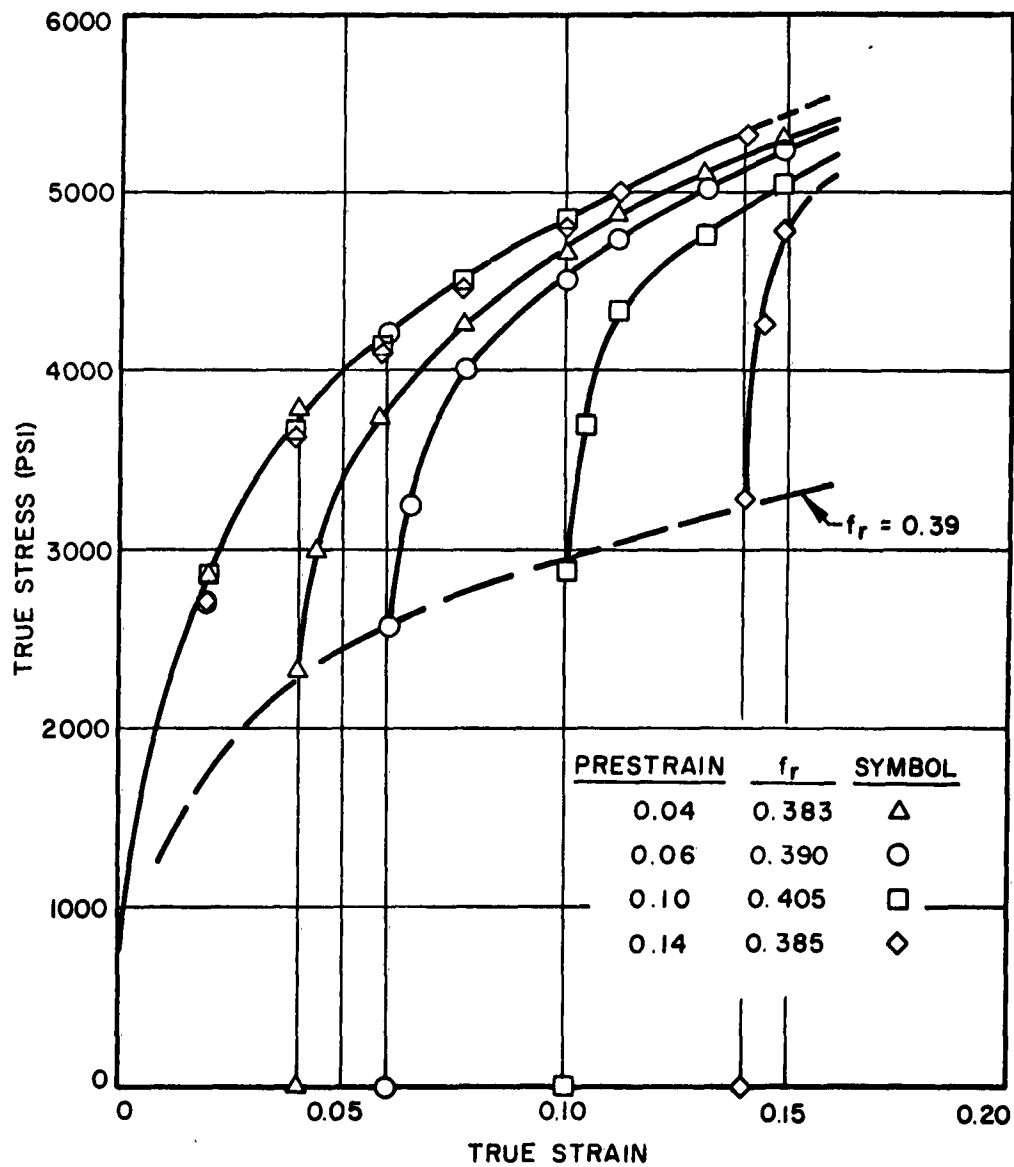


Fig. 15 Effect of Prestrain on the Stress-Strain Curve of 99.995-Percent Aluminum After Recovery for 41 Hr at 160°C

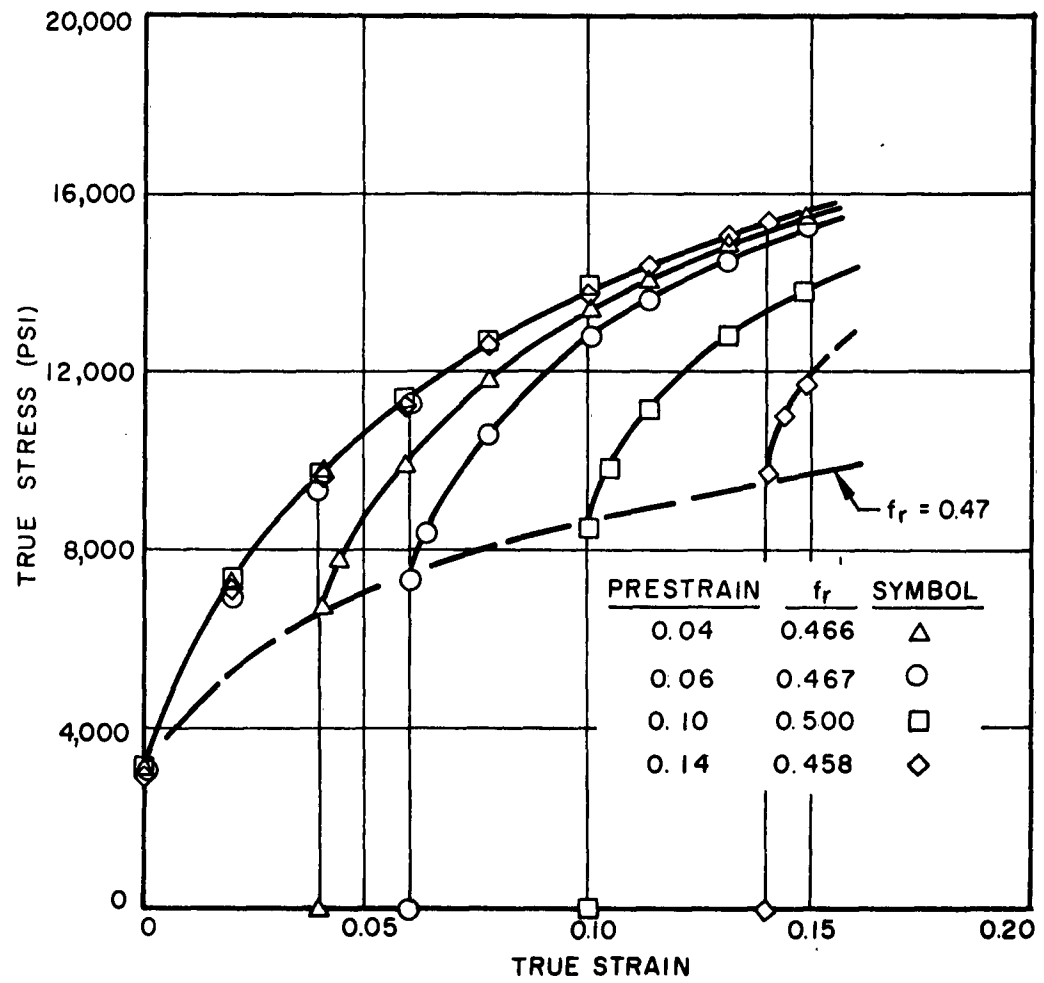


Fig. 16 Effect of Prestrain on the Stress-Strain Curve of Al-1%Mg Alloy After Recovery for 6.0 Hr at 160°C

for the different prestrain conditions. In fact, it is quite evident that after recovery the initial rate of strain hardening increases with increasing amounts of prestrain.

With reference to Fig. 16, a 6-hr recovery treatment at 160°C resulted in a constant f_r of about 0.47, independent of the amount of prestrain between 0.04 and 0.14. Also for the alloy the initial rate of strain hardening after recovery increases with increasing amounts of prestrain. Thus, the fractional recovery of initial flow stress appears to be independent of the amount of prestrain for both the high-purity aluminum and the Al-Mg alloy, over the range of prestrain studied.

3.3 RECOVERY OF HIGH-PURITY ALUMINUM UNDER CONCURRENT ELASTIC AND PLASTIC STRAIN

3.3.1 Recovery Under Concurrent Elastic Strain

Recovery tests were conducted on high-purity aluminum at 80° and 198°C under conditions of constant applied tensile stress which produced no measurable creep strain during the course of recovery treatment. These results are shown in Fig. 17. The solid lines indicate no-load recovery curves, and the data points show the results of recovery tests made under concurrent stresses of 690, 2500, and 3025 psi as indicated in the figure. It will be noted that the application of 2500 psi at 80°C produced no change in recovery rates. Furthermore, a concurrent stress of 690 psi produced no significant effect on recovery at 198°C. A stress of 3025 psi at 80°C, however, resulted in an appreciable acceleration of recovery throughout the range of recovery times investigated. This stress was found to produce measurable creep strain beginning at about 50 hr, as will be discussed in the next section. The possibility exists that some creep was occurring, but it was not measurable with the equipment employed; it is estimated that a creep strain of 0.001 or less could remain undetected.

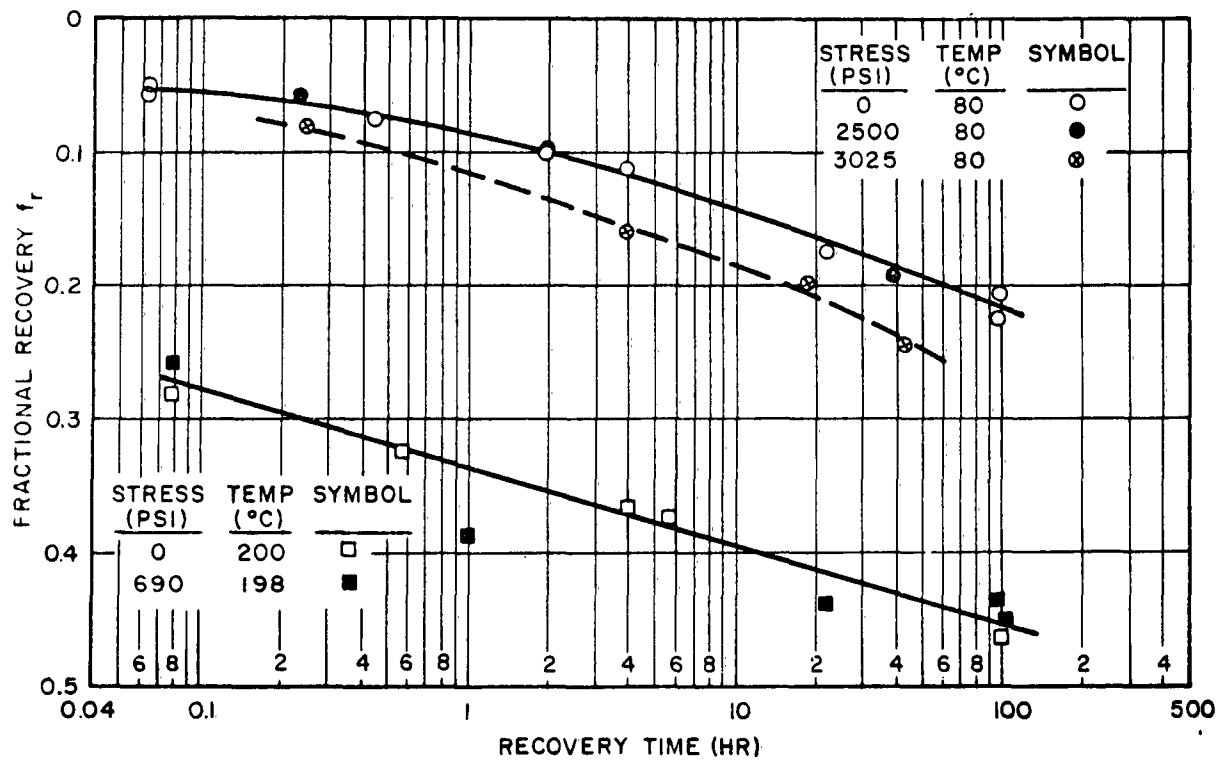


Fig. 17 Effect of Concurrent Stress for Which no Detectable Creep Occurred During Recovery of 99.995-Percent Aluminum at 80° and 198°C.

3.3.2 Recovery Under Concurrent Creep Straining

Creep tests conducted on high-purity aluminum prestrained to 0.10 true strain exhibited an initial stage of zero creep which was dependent upon the stress and temperature. The creep curves representing the highest concurrent strains used in this study are shown in Fig. 18. In each case, a period of time elapsed prior to the achievement of an appreciable creep rate. A series of recovery tests were conducted which were terminated at different points along the curves shown.

The effect of concurrent creep straining on the recovery of high-purity aluminum at 160° and 198°C under a stress of 2075 psi is shown in Fig. 19. The upper lines represent the no-load recovery curves. The numbers in parentheses refer to the creep strain which occurred in each test. These data clearly demonstrate that the recovery process is accelerated during concurrent creep strain. The fractional recovery is observed to have increased as much as 40 percent during creep straining in the strain range 0.03–0.05, and no significant acceleration of recovery at these temperatures was obtained prior to the onset of creep. These data allow a check on the equivalence of states in terms of the stress-strain curves after recovery, as two tests resulted in very similar fractional recovery: one after recovery for 1 hr at 198°C under 2075 psi had an f_r of 0.472, and the other after recovery for 14 hr at 160°C under the same stress had an f_r of 0.478. For comparison, the two tests are shown in Fig. 20 in terms of percent of true stress at 0.10 true strain. The good agreement of the two curves after different recovery treatments adds additional support to the concept that equivalent structural states exist at equal values of fractional recovery.

The effects of concurrent creep straining on recovery of high-purity aluminum were also studied at 3025 psi at 80°C and at 1385 psi at 198°C. These results are summarized in Fig. 21. As before, the numbers in parentheses refer to the creep strain achieved in each case; absence of a number indicates that no detectable creep occurred. It will be noted in Fig. 21a that, although accelerated recovery occurred after only 5 min at 80°C under a stress of 3025 psi, the onset of measurable creep, as indicated

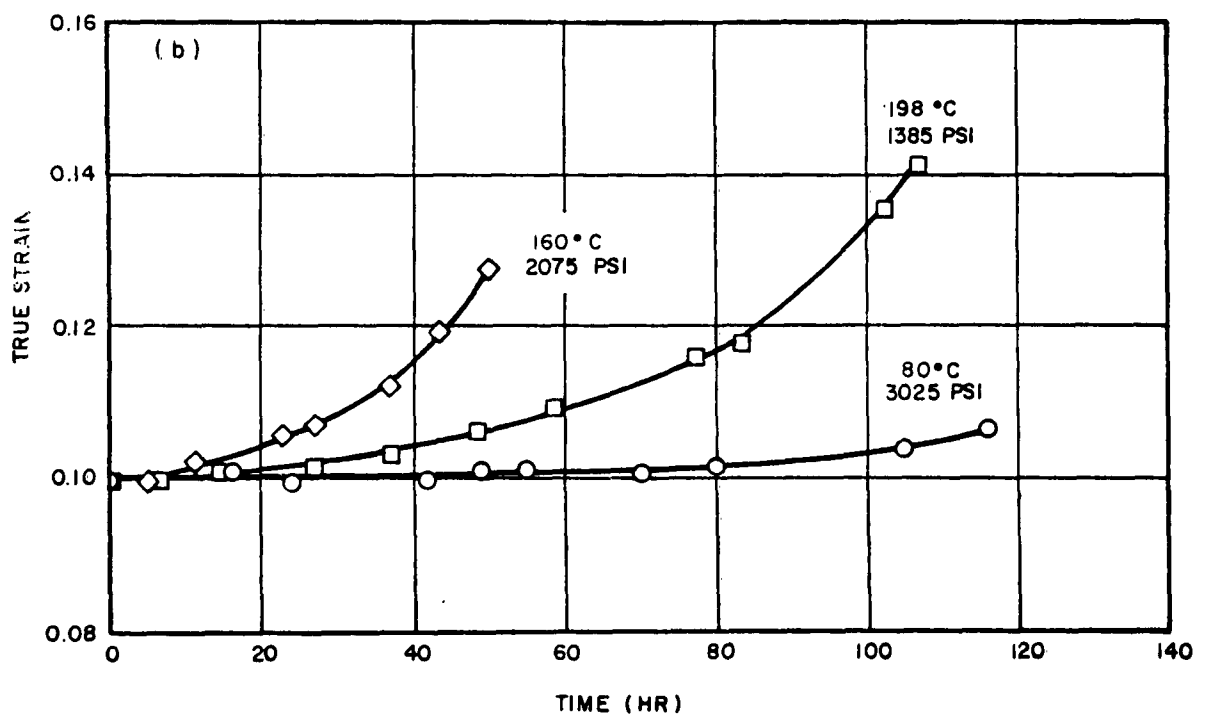
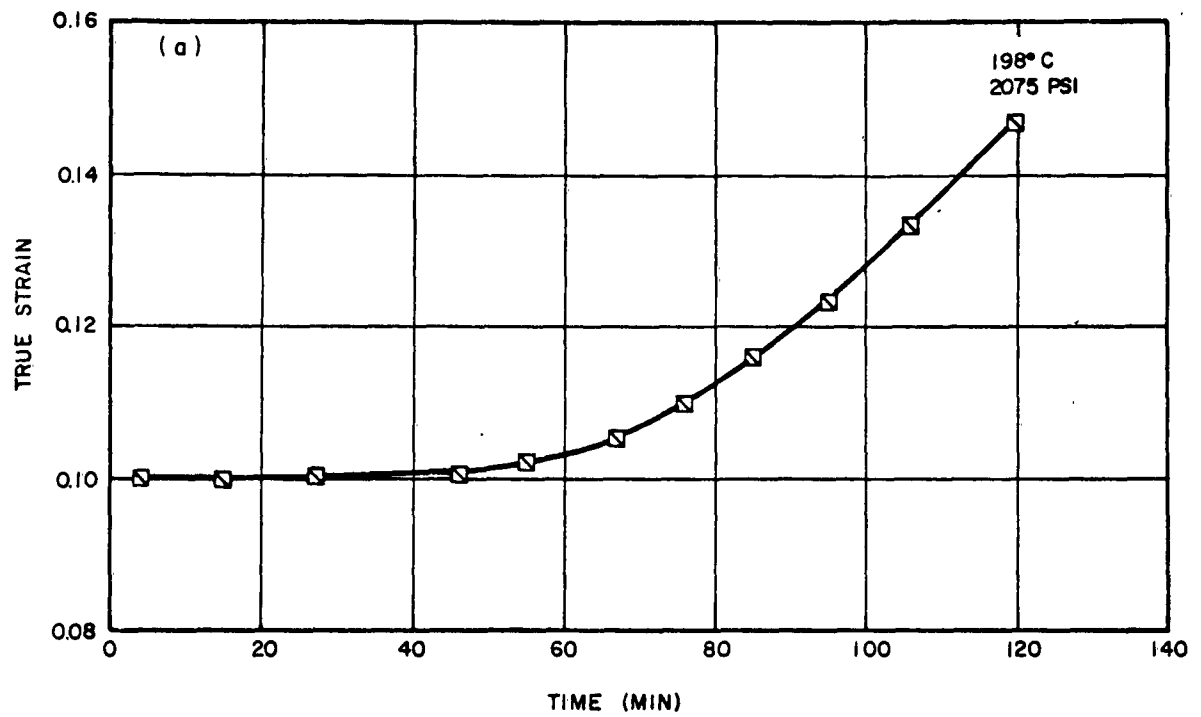


Fig. 18 Typical Creep Curves for 99.995-Percent Aluminum After Prestraining to 0.10 at Room Temperature

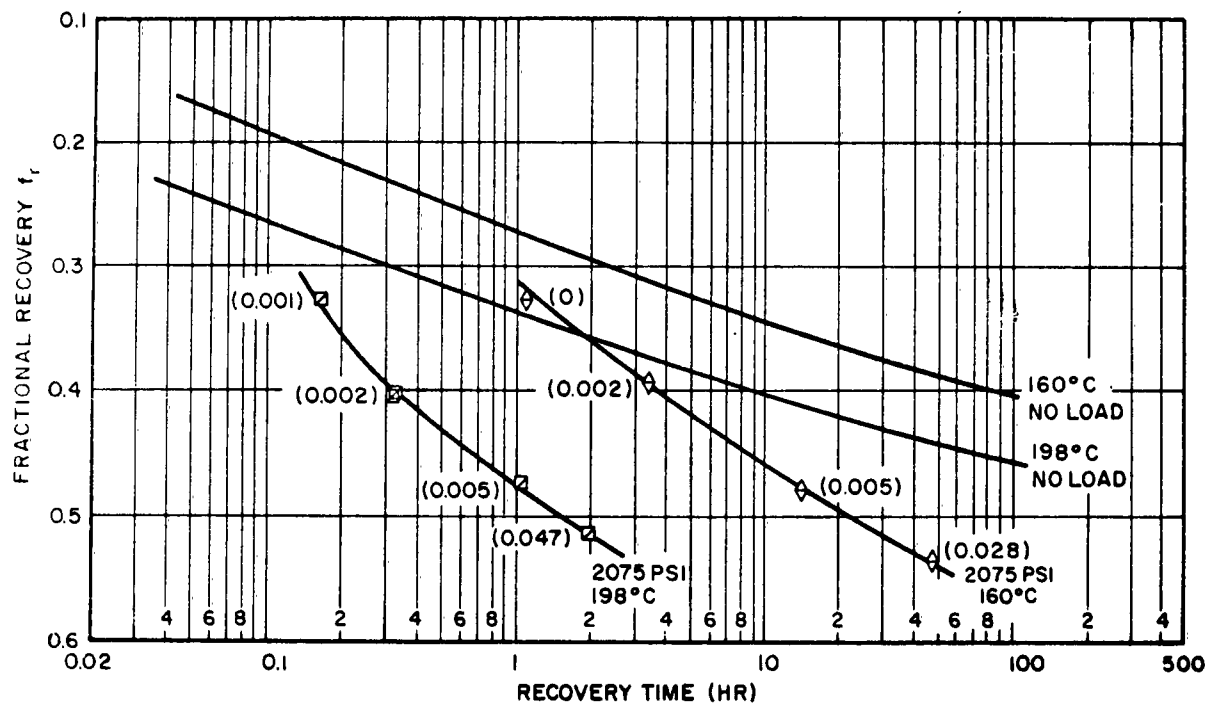


Fig. 19 Effect of Concurrent Creep Straining on Recovery of 99.995-Percent Aluminum at 160° and 198°C Under the Same Stress

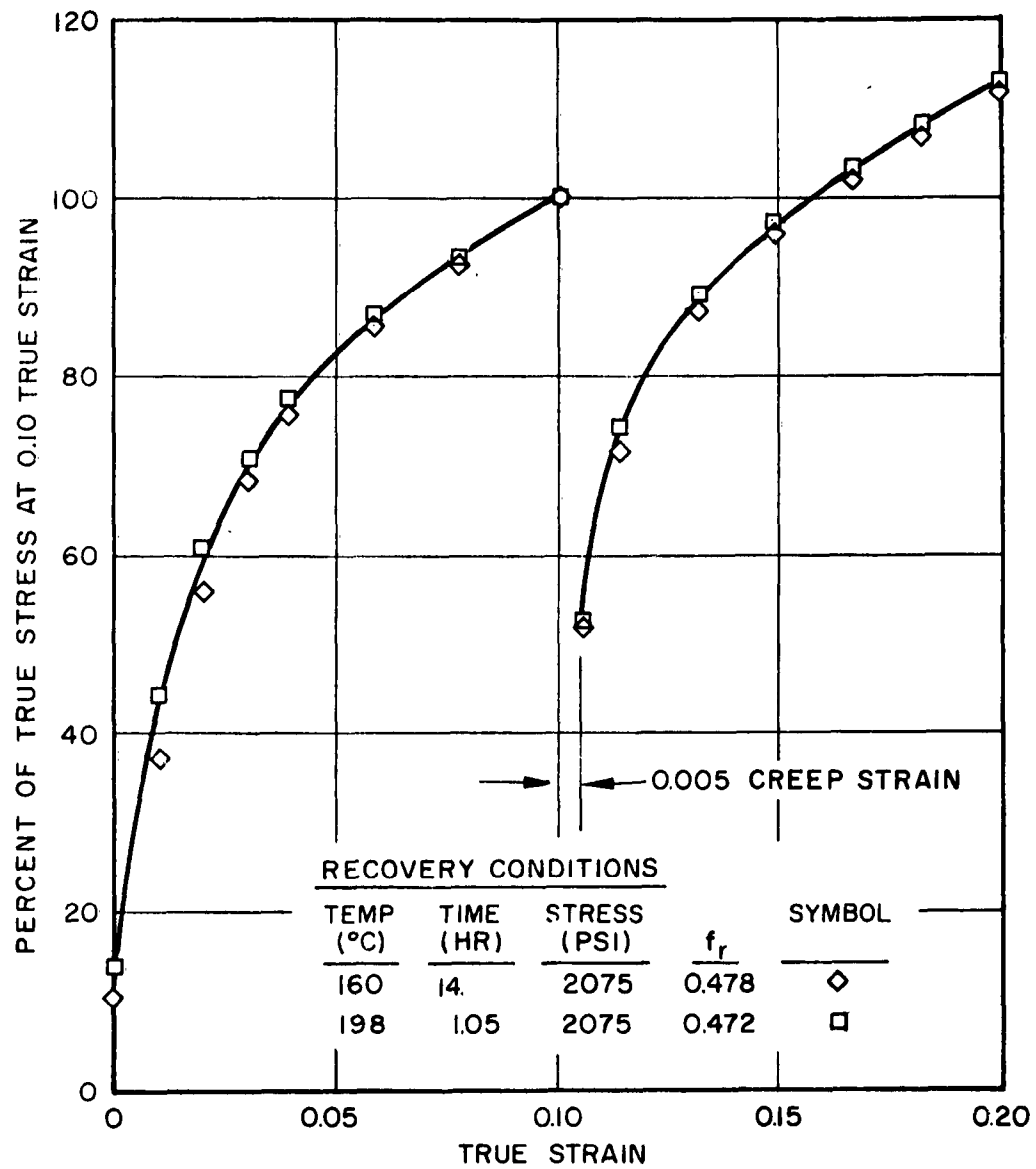


Fig. 20 Equivalence of Recovered States at Constant Fractional Recovery During Concurrent Creep Straining

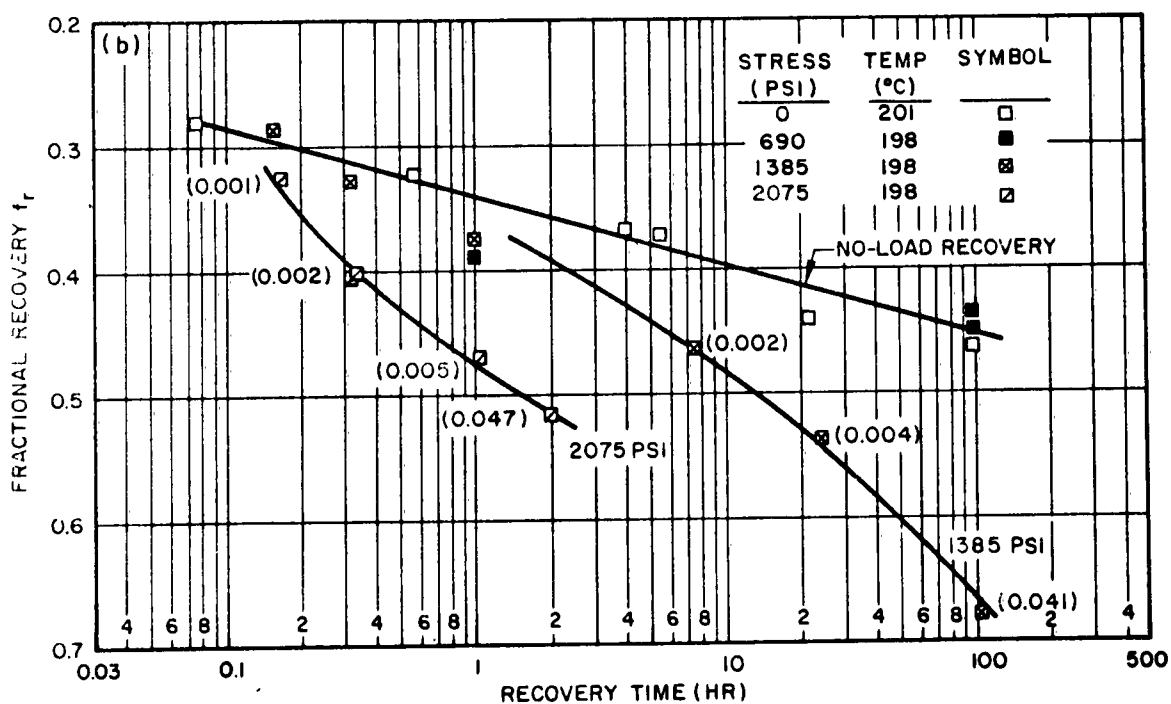
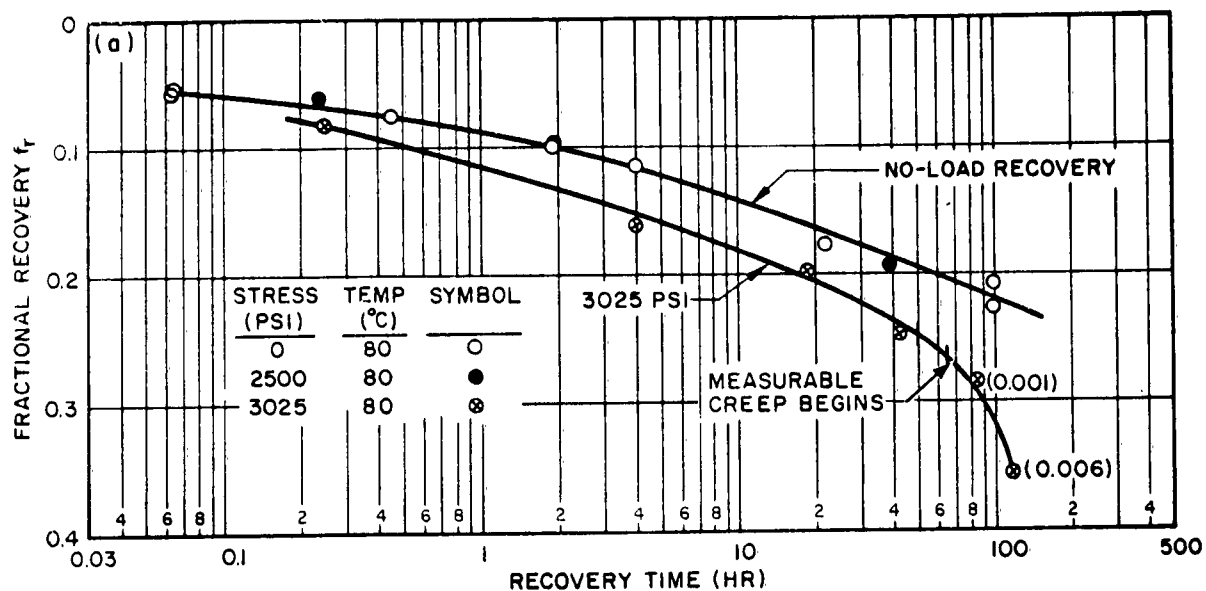


Fig. 21 Effect of Concurrent Creep Straining on Recovery of 99.995-Percent Aluminum at 80° and 198° C Under Various Stresses

in Fig. 18b, resulted in a sharp increase in the rate of recovery. As previously mentioned, small amounts of creep may be the cause of the accelerated recovery which occurred prior to 50 hr.

Figure 21b shows the effect of concurrent creep straining at 1385 psi and 2075 psi on the recovery of high-purity aluminum at 198°C. Again, it will be noted that appreciable acceleration of the recovery process at 198°C required creep straining. The form of the accelerated recovery curves does not appear the same, as the 1385-psi curve indicates a greater increase in fractional recovery at the higher creep strain. A possible explanation for this behavior will be discussed later.

Section 4

DISCUSSION

4.1 PREVIOUS INVESTIGATIONS

The results obtained by previous investigators suggest that no-load recovery of cold-worked, high-purity aluminum proceeds by polygonization and subgrain formation, followed at sufficiently high temperatures by subgrain growth. The following summary contains observations which are pertinent to this investigation:

- (1) Towner and Berger⁽⁷⁾ studied the recovery of cold-worked, high-purity aluminum single crystals and observed that:
 - Some substructure was present after deformation without heating. This observation was also made earlier by Perryman,⁽⁸⁾ on polycrystalline aluminum.
 - Heating to 400°, 500°, and 600°C caused only subgrain sharpening in the first 30 sec. Subgrain size at this point was dependent upon prestrain, being smaller for larger values of prestrain.
 - Following initial sharpening, subgrain growth occurred to some stable subgrain size which was dependent on temperature and prestrain. Higher temperatures caused larger subgrain sizes and greater prestrains caused smaller final sizes. This observation was also made by Perryman.⁽⁸⁾
- (2) Hirsch, Horne, and Whelan⁽⁹⁾ observed subgrains bounded by dislocation networks in thin films of aluminum by transmission electron microscopy. They observed that annealing of hammered films at 350°C caused sharpening of subgrains and removal of dislocations within the subgrains.
- (3) In addition to those observations previously mentioned, Perryman^(2, 8) observed that the subgrain size achieved during recovery of aluminum is

decreased by additions of magnesium. Furthermore, an addition of 0.1-percent magnesium was found to increase the rate of recovery of aluminum, but further additions had little effect.

- (4) Chaudron⁽¹⁰⁾ has reported that 99.9995-percent aluminum is virtually non-work-hardening at room temperature. This indicates that recovery is strongly controlled by impurity content, becoming more rapid with increasing purity.
- (5) Ball⁽¹¹⁾ has found that room-temperature flow stress for polycrystalline aluminum containing well-annealed substructure varies inversely as the square root of subgrain diameter.

It is apparent from the above observations that no-load recovery of cold-worked aluminum occurs in essentially two stages. The first stage is characterized by polygonization to form a preliminary subgrain structure which is dependent upon the previous amount of cold work. The second stage may be considered to consist of the perfection and growth of the subgrains to some final size which is dependent upon recovery temperature and impurity content. Furthermore, the work of Ball⁽¹¹⁾ indicates that the recovery fraction based upon flow stress as employed in this investigation will strongly reflect changes in subgrain size.

4.2 NO-LOAD RECOVERY OF HIGH-PURITY ALUMINUM

In light of the aforementioned experimental observations, the no-load recovery curves of Fig. 8 may be described as representing the decrease in strength, in terms of flow stress, which accompanies the processes of polygonization and subgrain growth. It will be noted that the no-load recovery curve at 80°C apparently exhibits two stages. The first stage is asymptotic to the zero recovery line and allows about 8-percent recovery of flow stress in the first hour. At that point, flow-stress recovery accelerates and enters a stage where fractional flow-stress recovery is increasing about linearly with the logarithm of recovery time. The 118°, 160°, and 201°C recovery curves and the linear portion of the 80°C recovery curve are all approximately

parallel during this stage, suggesting that the same process is taking place. It will be noted that each of the three upper curves must initially be asymptotic to the zero recovery line, since the degree of recovery must be zero at zero recovery time. Thus, it is concluded that the first stage of recovery exhibited at 80°C would be observed at the higher recovery temperatures if extremely short recovery times were within the realm of experimental practicability.

If the recovery behavior shown in Fig. 8 results from equivalent processes occurring at each temperature, a time-temperature correlation should be applicable. Based upon the equivalence of recovered states at equal values of fractional recovery, an activation energy for the recovery data shown in Fig. 8 may be calculated using the Dorn θ -parameter:⁽¹²⁾

$$\theta = te^{-Q/RT}$$

where

- t = recovery time
- Q = activation energy
- R = the gas constant
- T = the absolute temperature

The values of Q obtained for the no-load recovery of cold-worked, high-purity aluminum are shown in Fig. 8. These values show no significant trend with recovery or temperature; the average of these activation energies is 23,300 calories per mole.

A master plot of fractional recovery versus θ is shown in Fig. 22. The degree of correlation obtained strongly suggests that the process or processes occurring are thermally activated with an activation energy equal to about 23,300 calories per mole, and that equivalent recovered states are achieved at equal values of θ . This value of activation energy is in only fair agreement with the values reported by other investigators. Åström⁽¹³⁾ obtained a value of 28,000 calories per mole for recovery of

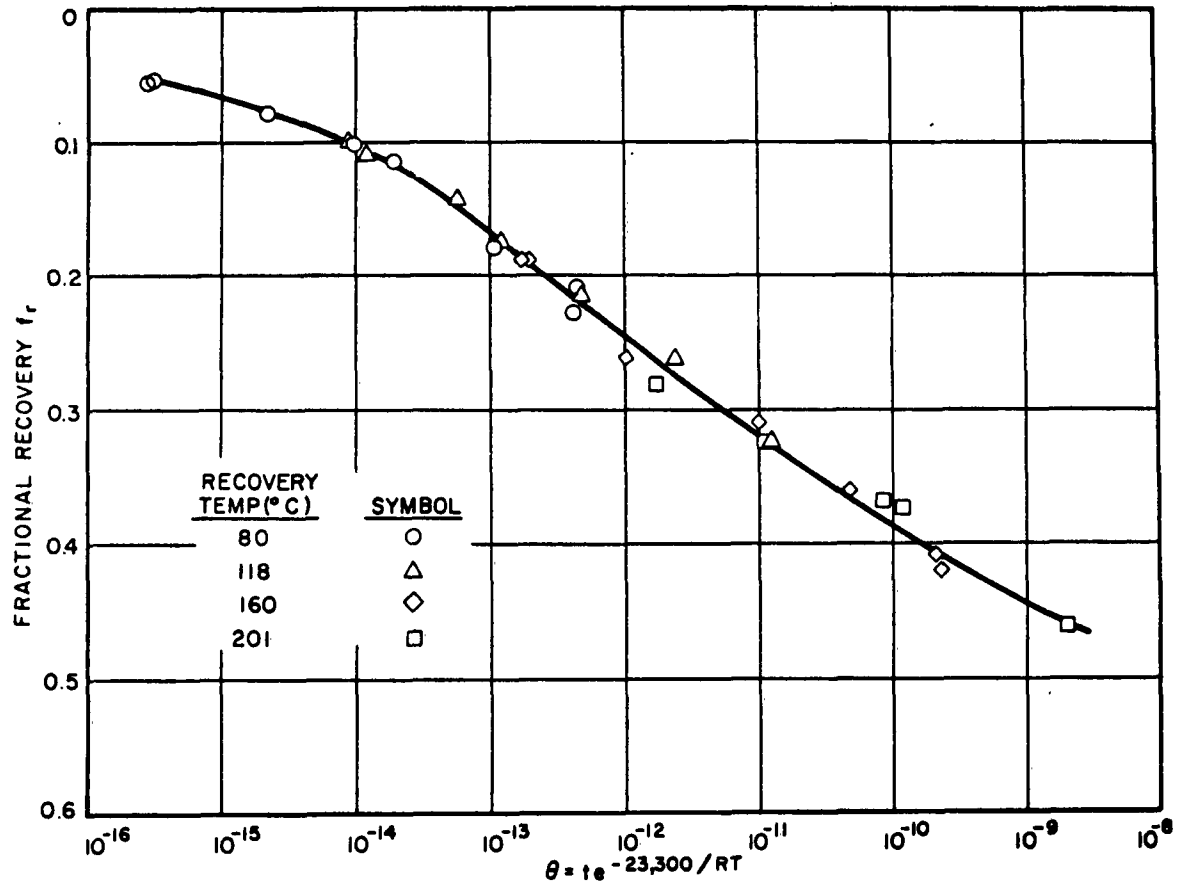


Fig. 22 Time Temperature Correlation of Fractional Recovery of 99.995-Percent Aluminum Using $\theta = t e^{-Q/RT}$

compressively strained aluminum up to about 180°C. Perryman⁽¹⁴⁾ employed hardness measurements on 20-percent cold-worked aluminum and reported an activation energy of 33,000 calories per mole, apparently at a temperature of about 350°C. At present, no explanation is offered for these reported differences. Based upon the present work, the activation energy for recovery of 10-percent cold-worked polycrystalline aluminum is concluded to be $23,300 \pm 2,000$ calories per mole in the temperature range 80°–200°C.

The possibility exists that the observed recovery behavior of high-purity aluminum below about 8-percent recovery is a process with an activation energy different from that reported above, since no data were available at times of less than about 5 min. It is suggested that this early stage corresponds to dislocation glide and climb to form an initial substructure, and that flow-stress recovery greater than 8 percent is a result of subgrain growth, with perhaps some additional increase in subgrain perfection. If this suggestion is correct, subgrain perfection and growth is still occurring after 100 hr at 200°C; that is, the final subgrain size has not yet been achieved, since the flow-stress recovery has not terminated. Subgrain studies are now in progress to determine the relationship between flow-stress recovery and polygonization and subgrain growth.

4.3 NO-LOAD RECOVERY OF Al-1% Mg ALLOY

The no-load recovery data obtained for cold-worked Al-Mg alloy are summarized in Fig. 12. The leveling-off of fractional recovery of flow stress suggests that a final stable structure has been reached at 118°, 160°, and 201°C. Previous investigators^(7,8) have reported that a stable substructure develops during recovery, wherein the stable size increases with increasing recovery temperature.

Additional evidence that very little structural change occurs after the fractional recovery reaches a plateau is shown by the fact that the entire stress-strain curves for Al-Mg specimens almost superimpose after recovery for 38, 99, and 476 hr, as presented in Fig. 11. As previously observed for high-purity aluminum, the 80°C recovery

curve for the Al-Mg alloy becomes asymptotic to the zero recovery line, possibly indicating an initial stage of recovery. A later stage of recovery at all four temperatures exhibits a linear increase in fractional flow-stress recovery with the logarithm of recovery time. As indicated earlier, however, the 201°C recovery curve is not parallel to the alloy curves obtained at the lower three temperatures but is parallel to the f_r versus $\log t$ curves obtained for the high-purity aluminum. Some change in recovery behavior apparently occurs between 160° and 201°C for Al-1% Mg alloys, possibly as a result of the interaction effects of magnesium atoms with dislocations and dislocation boundaries in this temperature range. It has been found that this effect drops off sharply between 160° and 200°C for Al-1% Mg.⁽¹⁵⁾ A further check on this possibility is provided by calculation of the activation energy for the recovery process in the Al-1% Mg alloy.

As indicated in the experimental results section, based upon equal values of fractional recovery, an activation energy value of 27,500 calories per mole was calculated for the Al-Mg alloy. Thus, the activation energy for recovery of cold-worked aluminum was observed to increase, owing to the addition of 1-percent magnesium. This value is in fair agreement with the value of 25,000 calories per mole reported by Perryman⁽¹⁴⁾ as measured by change in hardness of a similar material. Perryman, however, concluded that magnesium additions decreased the activation energy for recovery, having reported a value of 33,000 calories per mole for high-purity aluminum.

No detailed analysis of the value of 27,500 calories per mole obtained in this investigation will be attempted, but it is significant to note that Phillips, Swain, and Eborall⁽¹⁶⁾ obtained a value of 28,500 calories per mole for the strain aging of Al-3-1/2% Mg alloy between 50° and 150°C. This supports the suggestion that the same locking mechanism which accounts for strain-aging in Al-Mg alloys in the 50°-150°C range also inhibits the motion of dislocations and dislocation boundaries during the recovery process. Reported values for the activation energy for diffusion of magnesium in aluminum range from 26,800 to 38,500 calories per mole;⁽¹⁷⁾ however, the temperature range for these investigations was 365°-577°C, considerably higher than that used in the current study.

Subgrain studies are being conducted to determine the relationship between fractional flow-stress recovery and subgrain size and perfection for Al-1% Mg alloy.

4.4 RECOVERY UNDER CONCURRENT ELASTIC STRAIN

The effect of elastic strain on recovery behavior of metals is not clear from previous work. Tannenbaum and Kauzmann⁽¹⁸⁾ used creep rate before and after recovery treatment as a measure of recovery. They showed that recovery of zinc single crystals under a shear stress equal to 94 percent of the previous flow stress for a period of 100 min at 35°C gave the same degree of recovery as occurred under no-load conditions. Rinnovatore and Brown,⁽⁶⁾ using a fractional flow-stress recovery technique similar to that used in this investigation, found that shear stresses less than the yield point had no effect on the recovery behavior of zinc single crystals. Stresses greater than the yield point caused plastic strain and were reported to decrease the rate of recovery. Berghout⁽³⁾ used resistivity changes to measure recovery of polycrystalline copper wires at -1° and 180°C and reported that an applied stress of 17 kg/mm², causing elastic strain only, had an accelerating influence on recovery. No mention was made, however, of whether creep straining occurred after loading. The possibility of creep straining is especially significant at 180°C, where the work of Tietz and Dorn⁽¹⁹⁾ showed that 17 kg/mm² (24,200 psi) can cause continued creep in annealed OFHC copper.

As may be seen in Fig. 17, the recovery rate of 99.995-percent aluminum at 80°C is not affected by the application of a tensile stress of 2500 psi. However, the application of 3025 psi at 80°C is seen to produce an almost immediate increase in recovery which becomes greater with time. Two explanations for this increase are possible:

- Although no plastic straining was detected up to recovery times of 50 hr, the possibility remains that a small instantaneous strain and/or gradual creep strain of less than 0.001 in magnitude occurred.
- An elastic strain effect, as reported by Berghout,⁽³⁾ could have occurred.

The authors favor the first possibility for two reasons. First, the flow stress at 15-percent true strain of coarse-grained 99.997-percent aluminum at 80°C at slow strain rates was reported to be about 3000 psi.⁽²⁰⁾ Consequently, it is reasonable to expect some plastic straining at a stress of 3025 psi during recovery at 80°C from a 10-percent cold-worked state. At 198°C, the flow stress at 15-percent true strain is about 1150 psi,⁽²⁰⁾ suggesting that 690 psi would cause no plastic yielding. Second, if elastic strain alone has a significant effect on the recovery process, it is difficult to determine why no effect was detected at 2500 psi, whereas an appreciable effect was observed at 3025 psi. That is, it would not be expected that so sharp a threshold elastic strain would exist. Consequently, it is concluded that the flow-stress recovery of cold-worked polycrystalline high-purity aluminum between 80° and 200°C is not significantly affected by concurrent stresses which cause only elastic strains. It might be pointed out that the accelerated recovery of resistivity due to elastic strain shown by Berghout may be due to accelerated motion of vacancies to points of annihilation under stress gradients produced in the vicinity of grain boundaries and around dislocation networks. The effect of such a decreased concentration of vacancies would not have appreciable effects on the flow-stress measurements employed in this investigation unless it significantly influenced the polygonization and subgrain growth processes.

4.5 RECOVERY OF COLD-WORKED HIGH-PURITY ALUMINUM DURING CONCURRENT CREEP STRAINING

Previous investigations have given some indirect insight into the possible effects of concurrent plastic straining on the recovery of flow stress. Wood and Suiter⁽⁴⁾ have shown that a concurrent constant strain rate resulted in the growth of a subgrain structure in polycrystalline 99.98-percent aluminum to an equilibrium value which was related to the strain rate and temperature employed. Slower strain rates and higher recovery temperatures produced increased subgrain sizes. Also of interest is the study by Servi and Grant⁽²¹⁾ which describes the effect of creep straining under a constant stress upon the resulting substructure. These investigators showed that the subgrain diameter achieved during high-temperature creep of polycrystalline

high-purity aluminum was inversely proportional to the creep stress. The effect of higher constant strain rates may be considered to be equivalent to the effect of higher creep stresses in the development of equilibrium subgrain structures.

The results of Figs. 19 and 21 clearly show that for 10-percent prestrain and 80°–200°C recovery temperatures, creep straining accelerates the recovery process. Reference to these figures and subsection 4.4 also indicates that no appreciable influence was observed prior to the onset of creep straining.

The nature of the creep curves of Fig. 18 indicates that at zero recovery time the prestrained aluminum is capable of withstanding the applied stress. However, as recovery proceeds at these elevated temperatures, a recovered state is achieved for which the applied stress causes initial creep. Since equal fractional recovered states are achieved at equal values of θ , as indicated in the master f_r versus θ plot of Fig. 22, and applied stress has no effect on these recovered states prior to creep straining, it is to be expected that, for any given applied stress, creep would begin at some given fractional recovered state or given value of θ . After straining begins, however, a different activation energy may govern the recovery process. Based upon the equivalence of recovered states at equal values of f_r , demonstrated in Fig. 20 for concurrent creep straining, an activation energy was calculated for the concurrent strain recovery data of Fig. 19. The average value obtained was 25,000 calories per mole. This is somewhat higher than the value obtained for no-load recovery, but the difference may not be significant. A plot of fractional flow-stress recovery versus θ is now possible for both no-load and concurrent creep-strain recovery data, using only one value of Q . Figure 23a shows such a plot for $Q = 23,300$ calories per mole, the value obtained for no-load recovery. Good correlation is obtained, but there is a detectable separation between the 160° and 200°C concurrent creep recovery curves. As indicated in Fig. 23b, a plot using an overall Q value of 25,000 calories per mole is more successful for correlating the recovery data under concurrent creep straining, but the degree of correlation of the no-load data is slightly decreased. The activation energy for flow-stress recovery of polycrystalline 99.995-percent aluminum is thus

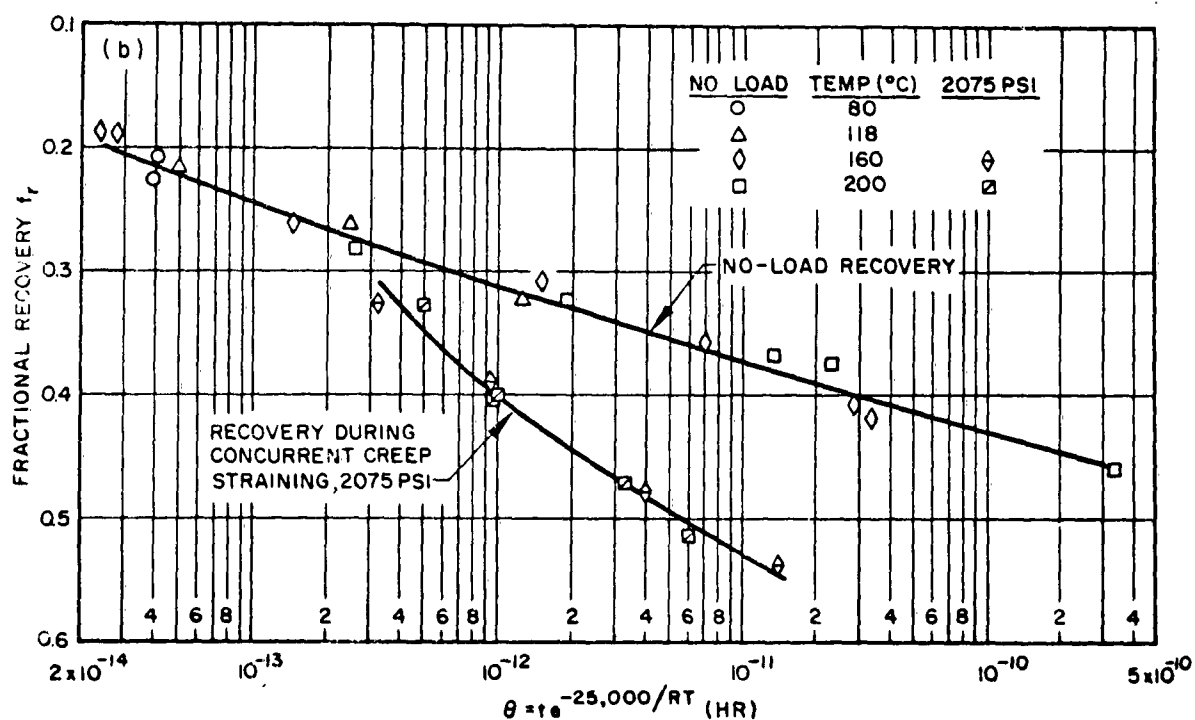
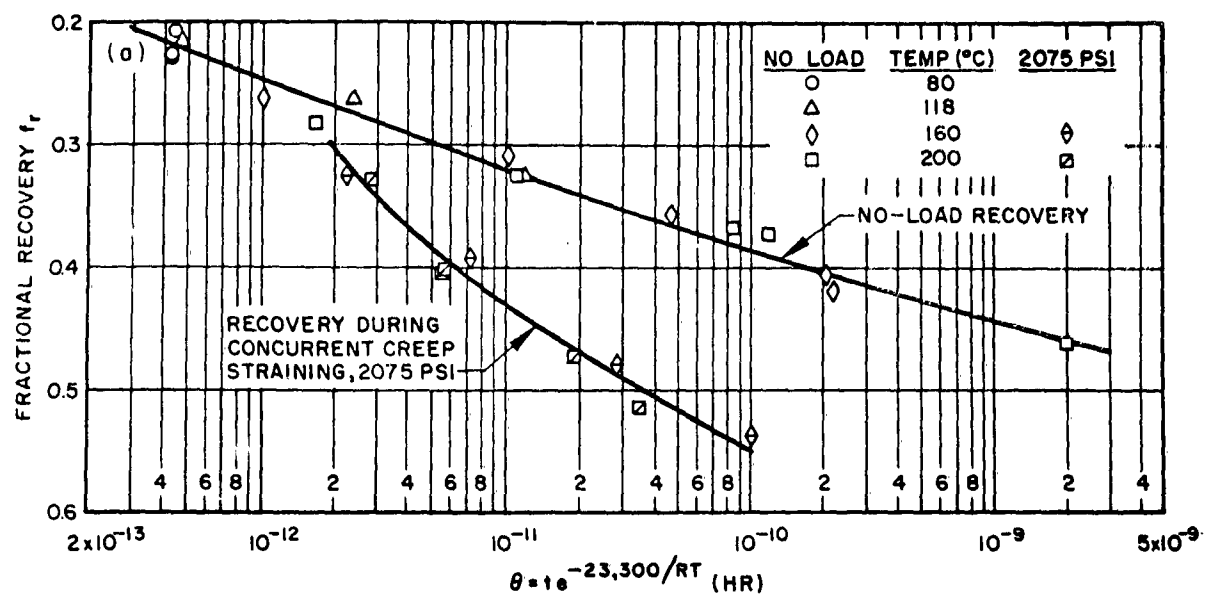


Fig. 23 Time-Temperature Correlations of Fractional Recovery for 99.995-Percent Aluminum for Both Conditions of No-Load and Concurrent Straining

not appreciably affected by concurrent creep straining over the range of fractional recovery and temperature investigated.

The effect of different creep stresses on acceleration of the flow-stress recovery may be ascertained from Fig. 21. Let us define the additional fractional recovery due to creep, Δf_c , as the fractional recovery under creep conditions at a given time and temperature minus that for no-load recovery at the same time and temperature. Using this parameter, the effect of creep strain upon acceleration of the fractional recovery is shown in Fig. 24. Three observations may be made from this plot:

- The initial creep straining (about 0.005) has the greatest effect upon Δf_c , this portion being rather insensitive to stress and temperature variations.
- The curves of Δf_c versus creep strain under the same creep stress of 2075 psi but different recovery temperatures of 160° and 200°C are essentially identical. This is consistent with the fact that the subgrain size achieved during high-temperature creep is dependent only on the creep stress.⁽²¹⁾ Furthermore, the correlation of Fig. 23 shows that the course of creep recovery at 2075 psi follows a given f_r versus θ path.
- Creep straining at 1385 psi produces a greater acceleration of recovery at 200°C than was obtained using 2075 psi. Since subgrain size has been observed to increase with decreasing creep stress, this behavior may be accounted for on the basis of subgrain growth toward a larger subgrain size under a creep stress of 1385 psi, leading to a larger final value of f_r and thus Δf_c . No further comments can be made regarding the results for creep recovery at 80°C and 3025 psi, since a strain of only 0.006 was achieved.

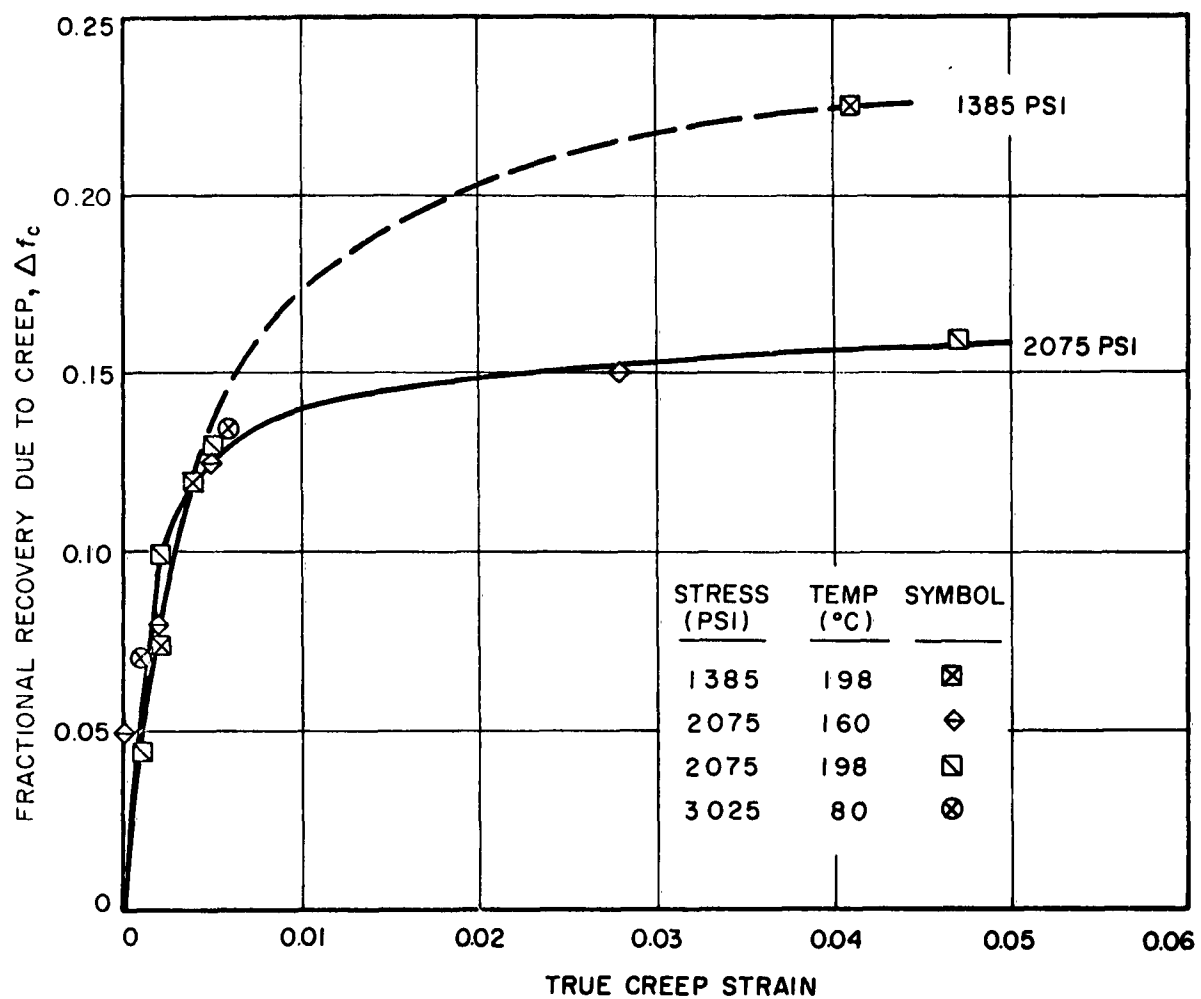


Fig. 24 Fractional Recovery Due to Creep as a Function of Creep Strain for 99.995-Percent Aluminum

Section 5

CONCLUSIONS

The following conclusions can be drawn:

- The activation energy for no-load recovery of polycrystalline 99.995-percent aluminum over a temperature range of 80° to 200°C was found to be 23,300 \pm 2,000 calories per mole.
- The addition of 1-percent magnesium to the high-purity aluminum was found to increase the activation energy for recovery to 27,500 calories per mole.
- The application of tensile stresses which caused only elastic strains had no significant effect upon the flow-stress recovery of polycrystalline 99.995-percent aluminum over a temperature range of 80° to 200°C.
- Flow-stress recovery of the 99.995-percent aluminum was accelerated by concurrent creep straining. At a given temperature, the increased recovery due to creep straining was greater under lower creep stresses for equal amounts of creep strain above about 0.005.
- The activation energy for flow-stress recovery during creep straining of the 99.995-percent aluminum between 160° and 200°C was found to be about 25,000 calories per mole. This value, only slightly greater than that found for no-load recovery, is not considered a significant difference.
- During a constant recovery treatment, the fractional flow-stress recovery values were found to be independent of prestrain values between 0.04 and 0.14 for both the 99.995-percent aluminum and the Al-1% Mg alloy.

Section 6
REFERENCES

1. E.C.W. Perryman, "Recovery of Mechanical Properties," Creep and Recovery, Cleveland, American Society for Metals, 1957, p. 111
2. E.C.W. Perryman, "Recovery of Cold-Worked High Purity Al-Mg Alloys," Trans. A.I.M.E., Vol. 206, Oct 1956, p. 1247
3. C.W. Berghout, "The Influence of Elastic Strains on the Recovery of Cold-Worked Copper," Acta Met., Vol. 6, 1958, p. 613
4. W.A. Wood and J.W. Suiter, "Stress-Recovery in Aluminum," J. Inst. Metals, Vol. 80, 1952, p. 501
5. O.D. Sherby, A. Goldberg, and J. E. Dorn, "Effect of Prestrain Histories on the Creep and Tensile Properties of Aluminum," Trans. Am. Soc. Met., Vol. 46, 1954, p. 681
6. J. V. Rinnovatore and Norman Brown, "Effect of Stress Upon the Recovery and Effect of Negative Strain Upon the Yield Point of Zinc Single Crystals," Trans. A.I.M.E., Vol. 218, 1960, p. 777
7. R.J. Towner and Alfred Berger, "X-ray Studies of Polyganization and Sub-grain Growth in Al", Trans. A.I.M.E., Vol. 218, No. 4, Aug 1960 p. 611
8. E.C.W. Perryman, "Observations on the Structural Changes Accompanying Recovery in Superpurity Al," Acta Met., Vol. 2, Jan 1954, p. 26
9. P.B. Hirsch, R.W. Horne, and M.J. Whelan, "Direct Observations of the Arrangement and Motion of Dislocations in Aluminum," Dislocations and Mechanical Properties of Crystals, New York, Wiley, 1957, p. 92
10. G. Chaudron, Congress International de l'Aluminum, Vol. 1, Paris, Jun 1954 p. 179

11. C.J. Ball, "The Nature and Effect of Substructure in Polycrystalline Aluminum," Dislocations and Mechanical Properties of Crystals, New York, Wiley, 1957, p. 353
12. O.D. Sherby and J.E. Dorn, "Creep Correlations in Alpha Solid Solutions of Aluminum," Trans. A.I.M.E., Vol. 194, 1952, p. 959
13. H.V. Åström, "Isothermal Measurements on the Release of Energy Stored in Cold-Worked Aluminum," Acta Met., Vol. 3, 1955, p. 508
14. E.C.W. Perryman, "Recrystallization Characteristics of Superpurity Base Al-Mg Alloys Containing 0 to 5% Mg," Trans. A.I.M.E., Vol. 203, 1955, p. 369
15. O.D. Sherby, R.A. Anderson, and J.E. Dorn, "Effect of Alloying Elements on the Elevated Temperature Plastic Properties of Aluminum," Trans. A.I.M.E., Vol. 191, 1951, p. 643
16. V.A. Phillips, A.J. Swain, and R. Eborall, "Yield Point Phenomena and Stretcher-Strain Markings in Aluminum-Magnesium Alloys," J. Inst. Metals, Vol. 81, 1952-53, p. 625
17. C.J. Smithells, Metals Reference Book, Vol. 2, New York, Interscience Publishers, 1955, p. 552
18. M. Tannenbaum and W. Kauzmann, "Studies on the Creep Recovery and Annealing of Zinc Single Crystals," J. Appl. Phys., Vol. 25, No. 4, 1954, p. 451
19. T. E. Tietz and J. E. Dorn, "Creep of Copper at Intermediate Temperatures," Trans. A.I.M.E., Vol. 206, 1956, p. 156
20. T.A. Trozera, O.D. Sherby, and J.E. Dorn, "Effect of Strain Rate and Temperature on the Plastic Deformation of High Purity Aluminum," Trans. Am. Soc. Met., Vol. 49, 1957, p. 173
21. I.S. Servi and N.J. Grant, "Structure Observations in Aluminum Deformed in Creep at Elevated Temperatures," Trans. A.I.M.E., Vol. 191, 1951, p. 917

<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p>	<p>UNCLASSIFIED</p>	<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p>	<p>UNCLASSIFIED</p>
<p>(over)</p> <p>reaction was also studied to determine the possibility of catalysis by ultra-violet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>	<p>(over)</p> <p>reaction was also studied to determine the possibility of catalysis by ultra-violet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>

<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p> <p>(over)</p>	<p>UNCLASSIFIED</p>	<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p> <p>(over)</p>	<p>UNCLASSIFIED</p>
<p>reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a <u>D.P.</u> of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>	<p>reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a <u>D.P.</u> of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>
	<p>UNCLASSIFIED</p>		<p>UNCLASSIFIED</p>

<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p>	<p>UNCLASSIFIED</p>	<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p>	<p>UNCLASSIFIED</p>
<p>(over)</p> <p>reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>	<p>(over)</p> <p>reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>

<p>UNCLASSIFIED</p>	<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p>	<p>UNCLASSIFIED</p> <p>(over)</p>
<p>UNCLASSIFIED</p>	<p>reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>
<p>UNCLASSIFIED</p>	<p>THE DOW CHEMICAL COMPANY, Midland, Mich., PHENYLENE SULFIDE POLYMERS, by Robert W. Lenz, Carl E. Handlovits and Harry A. Smith, June 1961. 63 p. incl. figs. and tables. (Project 7340; Task 73404) (WADD TR 61-139) (Contract AF 33(616)-7251) Unclassified report</p> <p>Investigations were carried out toward the preparation of high molecular weight polyphenylene sulfide for high temperature applications. The reactions of sodium thiophenoxide with p-dibromo- and p-diiodobenzene were studied in some detail to collect information on the rates of these reactions and on the nature and extent of secondary reactions. An analogous</p>	<p>UNCLASSIFIED</p> <p>(over)</p>
<p>UNCLASSIFIED</p>	<p>reaction was also studied to determine the possibility of catalysis by ultraviolet light. Convenient procedures were developed for the preparation of p-fluoro-, p-bromo-, and p-iodothiophenol. Pure, anhydrous salts of these compounds were prepared and polymerized both in the melt and in solution. Linear polyphenylene sulfide, prepared from sodium p-bromothiophenoxide and having a D.P. of approximately 20, was found to be kinetically stable in nitrogen and air up to 400°C.</p>	<p>UNCLASSIFIED</p>